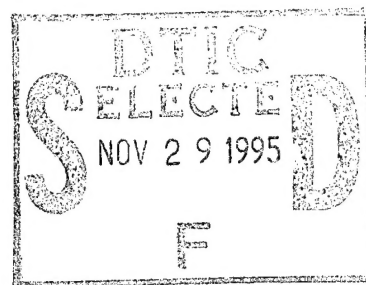


## Analysis of Asbestos Uniformity

15 November 1995



Prepared by

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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

 *may, USAF*  
J. Cole, MAJ, USAF

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## Analysis of Asbestos Uniformity

A. H. Zimmerman and M. V. Quinzio

Recent concerns about the effects of various contaminants on the performance of  $\text{NiH}_2$  cells has created interest in evaluating the quantities of various contaminating materials in asbestos separator material. References 1 and 2 have quantified the amounts of mineral contaminants for random samples of asbestos, based on the reactivity of the asbestos towards KOH electrolyte. This work evaluates the amount of contaminating materials present based on reactivity towards both acid and basic solutions, and in addition correlates the amounts of reactive components with position across the asbestos roll. These results are then interpreted to suggest why some cell lots experience greater problems from contaminants than other lots, and why the problems tend to be highly variable within a given lot.

The separator samples analyzed here have also been utilized in assembling small  $\text{NiH}_2$  test cells for the purpose of evaluating the effects of contaminants on the performance of the cells. A critical portion of this test effort is the evaluation of the level and amounts of contaminants present in each separator used in these test cells. These data are provided here for later use in correlations with the performance of these test cells.

The samples used in this study are numbered 1-12, and were cut at uniform intervals of three inches from a strip obtained from a three-foot wide roll of asbestos. This roll of asbestos has been used immediately prior to this sampling to construct a nickel hydrogen cell lot which had given quite significant performance problems that were attributed to contaminants. Thus there was some reason to believe that both variable and high levels of contaminating substances might be found in this asbestos sample. From each of these 12 samples of asbestos, two 1 in<sup>2</sup> samples were cut from adjacent areas for analysis. These samples are labelled 1-12A and 1-12B. The A samples were subjected to 0.15 M nitric acid for 4 hr at 70 deg C. Following this treatment, the asbestos residue was filtered from the solution, dried, and weighed. The filtered solution was then brought to pH 7-8 using a sodium acetate buffer, then treated with an excess of 8-hydroxyquinolinol (HQ). This reagent precipitates a wide range of metals as a chelate. EDAX of the chelate precipitates was performed to provide an indication of the differences in composition of the material extracted from the asbestos samples by dilute nitric acid.

Table 1 indicates the starting weight of each sample used in the analysis described in the above paragraph, followed by the weight lost and that precipitated as the HQ chelate. Since these chelates are presumably resulting from a mixture of metals (all having differing weights), no attempt has been made to calculate the proportion of the chelate weight resulting from the leached metals.

Table 1. Acid Extraction of Asbestos Samples

ID	Initial Weight (g)	Weight Loss (g)	Weight Loss (%)	HQ Chelate Wt (g)
1A	0.1109	0.0496	44.7	0.0092
2A	0.1113	0.0476	42.8	0.0069
3A	0.1118	0.0456	40.8	0.0067
4A	0.1118	0.0464	41.5	0.0084
5A	0.1112	0.0527	47.4	0.0083
6A	0.1116	0.0502	45.0	0.0085
7A	0.1113	0.0520	46.7	0.0102
8A	0.1116	0.0483	43.3	0.0171
9A	0.1112	0.0402	36.2	0.0113
10A	0.1143	0.0421	36.8	0.0081
11A	0.1105	0.0401	36.3	0.0114
12A	0.1100	0.0500	45.5	0.0086

Figure 1 provides a histogram of the weights of metal chelates found in these 12 samples. The results in Fig. 1 indicate that a region in the asbestos roll around sample 8 has a significantly different and higher level of contamination than does the rest of the roll. Figure 2 correlates the weight loss by the asbestos samples during the acid extraction with the weight of chelate precipitate obtained from each sample. For a single contaminating material in the asbestos, there should be a direct correlation between the weight loss and the weight of chelate. However, Fig. 2 indicates two discrete proportionalities. Samples 8A, 9A, and 11A fall on one line, with all the remaining samples falling on a lower line having approximately half the slope of the first line (except for sample 10A, which lies between these two lines). The most straightforward interpretation of Fig. 2 is that the asbestos roll between samples 8A and 11A contained an additional contaminating substance that was not significantly present elsewhere. This is a region approximately 30% of the width of the roll.

To determine the chemical composition of the contaminating substances suggested by Fig. 2, EDAX analysis of the chelates in Table 1 were performed. Table 2 presents the results of these chelate analyses. Each analysis is the average of several EDAX analyses performed on macroscopic (about 1mm) particles of the chelate, except where noted. For several of the samples small particles of a unique shape were analyzed as noted.

Table 2. EDAX Analyses of Acid-Extracted Chelates

Sample	% Iron	% Si	% Al	% Mg	% Mn	% Ca	% K
1A	72.22	5.22	21.18	0.00	1.38	0.00	0.00
2A	70.70	6.31	20.83	0.00	2.16	0.00	0.00
3A	74.87	3.68	20.16	0.00	1.29	0.00	0.00
4A	73.16	4.85	18.95	0.00	3.04	0.00	0.00
5A	72.48	6.11	17.27	0.00	4.13	0.00	0.00
6A	70.73	3.85	23.26	0.00	2.16	0.00	0.00
7A	55.82	4.25	25.64	0.00	4.20	0.00	10.10
8A	12.18	0.00	0.00	86.63	1.24	0.00	0.00
9A	47.30	5.14	9.00	35.50	3.02	0.00	0.00
10A	72.50	5.84	18.31	0.00	3.36	0.00	0.00
11A	75.26	4.77	12.74	19.12	5.49	0.00	0.00
12A	70.60	4.74	19.32	0.00	5.34	0.00	0.00
2A Large Particle	0.00	98.28	1.72	0.00	0.00	0.00	0.00
5A Large Particle	6.69	0.00	0.00	75.99	0.00	17.33	0.00
10A rod	0.00	3.06	0.00	96.94	0.00	0.00	0.00
11A Granule	69.50	4.72	15.18	2.02	8.57	0.00	0.00
11A rod	4.88	58.84	0.00	36.10	0.18	0.00	0.00

The EDAX scans upon which Table 2 is based are included in Appendix 1. The percentages reported in Table 2 are simply based upon the peak heights seen in the EDAX scans.

Table 2 indicates that the asbestos contains a relatively uniform background level of contaminants consisting of iron and aluminum, with small amounts of manganese and silicate showing up as well. In addition to these contaminating elements, for samples 8-11 an additional contaminant that was very high in magnesium is present. This additional contaminating material is the reason for the upper line in these samples in Fig. 2. It is likely that this magnesium contaminant is a separate phase from the magnesium silicate that is the major constituent of asbestos, since the normal magnesium silicate phase in asbestos is quite stable to acid.



In addition to the magnesium containing contaminant indicated in samples 8-11, Table 2 indicates a number of other spot contaminants distributed more-or-less randomly across the samples. In sample 2A some chelate particles containing only aluminum and predominantly silicon were found. In sample 5A chelate particles containing predominantly magnesium and calcium were found. In sample 7A unusually high levels of aluminum were found along with significant amounts of potassium. In samples 10A and 11A rod-shaped and granule-shaped chelate particles were found that contained either large levels of magnesium, silicon, or manganese. In sample 12A one EDAX analysis indicated quite high levels of silicon. Presumably, the high levels of silicon that were not associated with other metals (such as in the last entry in Table 2) are due to significant silicate or silica levels that either were not associated with a metal that formed a chelate with HQ, or the metals remained in the asbestos in the form of an insoluble reaction product.

To determine which of the contaminants detected in the asbestos samples were dissolved in KOH electrolyte, and thus might affect nickel hydrogen cell performance, samples 1B to 12B were exposed to 38% KOH at 40 deg C for 2 weeks. Following this exposure, these samples were rinsed and filtered to determine the weight loss in KOH. The KOH rinsed from these samples was analyzed for insoluble silicates by evaporating to dryness after neutralization with HCl. The dry crystals were then dissolved in DI water, and insoluble silicates (or other

Table 3. Weight Loss and Materials Extracted In KOH from Asbestos

Sample ID	Init Wt (g)	% Weight loss	KCl Wt (g)	Silicate Res %	% Calcium Sulfate
1B	0.1102	3.99	1.2535	2.18	0.37
2B	0.1117	3.58	0.7470	1.16	0.05
3B	0.1131	3.36	0.8473	2.12	0.05
4B	0.1094	3.56	0.6061	1.55	0.11
5B	0.1135	2.11	0.7829	1.50	0.36
6B	0.1127	2.66	0.6642	1.60	0.46
7B	0.1131	3.71	0.7477	0.97	0.36
8B	0.1112	3.24	0.7318	0.90	0.37
9B	0.1130	4.16	0.5986	2.21	0.36
10B	0.1149	3.79	0.7209	1.31	0.61
11B	0.1100	1.36	0.7053	1.45	0.42
12B	0.1140	3.95	0.8761	0.96	0.41



compounds) were filtered from the solution and weighed. The weight of this largely silicate residue is reported in Table 3 along with the weight loss of the asbestos resulting from exposure to KOH, both as percentages of the original asbestos weight.

The weight losses in KOH (Table 3) are much lower than those reported in Table 1 due to exposure to dilute nitric acid. To determine precisely how the KOH had affected the asbestos, the samples of Table 3 were extracted with dilute nitric acid following their KOH exposure. The nitric acid solution filtered from each sample was then treated with HQ as described previously to give a chelate from any metals extracted from the asbestos. The results from this chelate analysis are indicated in the histogram of Fig. 1, as well as in Table 4 along with the weight loss from each of these KOH treated samples in nitric acid.

Table 4. Acid Analysis of Asbestos After KOH Exposure

Sample ID	Sample Wt (g)	Wt Loss (g)	% Wt Loss	Chelate Wt (g)
1B	0.1058	0.0342	31.0	0.0039
2B	0.1077	0.0292	26.1	0.0039
3B	0.1093	0.0375	33.2	0.0048
4B	0.1055	0.0406	37.1	0.0050
5B	0.1111	0.0415	36.6	0.0049
6B	0.1097	0.0319	28.3	0.0033
7B	0.1089	0.0414	36.6	0.0053
8B	0.1076	0.0460	41.4	0.0057
9B	0.1083	0.0438	38.8	0.0050
10B	0.1106	0.0446	38.8	0.0044
11B	0.1085	0.0489	44.5	0.0050
12B	0.1095	0.0487	42.7	0.0051

The results in Table 4 indicate that for samples 1 to 7 a significantly lower weight loss was observed for the acid rinse following the KOH treatment, relative to Table 1 for a direct acid rinse. Samples 8-10, on the other hand have very similar percentage weight losses. Since Figure 1 indicates that the KOH treatment is actually removing significant material from the asbestos in samples 8 to 11, this means that the KOH must be replacing anions with hydroxide ions through exchange reactions. Figure 3 illustrates this kind of exchange process by showing the difference in percentage weight loss during an acid rinse and the percentage weight loss during an acid rinse following KOH treatment. In Fig. 3 a positive value for a given sample means that acid extractable material was lost during the KOH treatment, while

a negative indication means that the KOH treatment actually added acid extractable material to the sample. This clear difference between samples 1-7 and samples 8-12 is expected to be due to anionic exchange with hydroxide ions in KOH. These results suggest that in samples 8 to 12 silicate, sulfate, or phosphate containing materials exist that can react with KOH to solubilize the anionic species, while leaving the metallic species as an insoluble hydroxide. Of course, these hydroxide species are readily dissolved by dilute nitric acid during a subsequent acid rinse.

The correlation between the weight losses in Table 4 and the weights of HQ chelate found is presented in Fig. 4. There is clearly a correlation between weight loss and chelate weight. It seems particularly noteworthy that samples 8 to 12 are the highest samples in this correlation, and exhibit considerably more scatter than do the other samples. This is again consistent with a significantly different composition in samples 8-12 relative to samples 1-7 with regard to stability towards KOH electrolyte.

The histogram (Fig. 3) of the difference in weight loss during the acid rinse before and after treatment of the asbestos samples with KOH provides a clear indicator for the quality of the asbestos relative to exchange reactions with KOH. Fig. 3 clearly indicates that one side of the asbestos roll has a significantly differing composition from the other side of the roll. Considering the EDAX analysis results reported above, the half of the roll involving samples 8-12 would be significantly more likely to introduce soluble contaminants into the electrolyte of a nickel hydrogen cell. An analysis of the kind reported here could, in principle, be used to identify portions of the asbestos roll that should not be used for building nickel hydrogen cells.

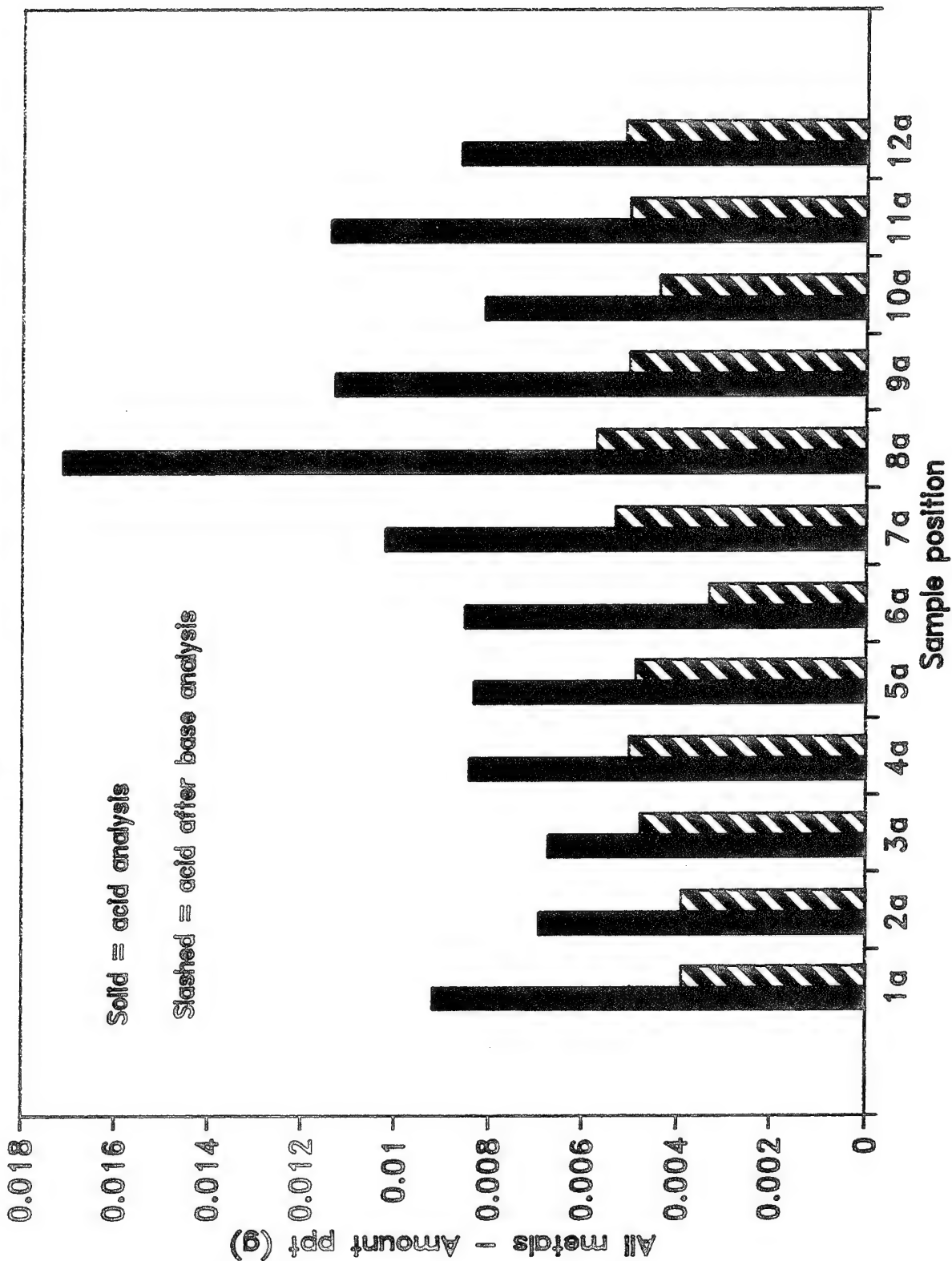
The results reported here suggest a need for several tests to support the idea that components of asbestos which can readily undergo exchange reactions with KOH electrolyte may be responsible for poor performance of nickel hydrogen cells. These tests will compare the performance of test nickel hydrogen cells made with asbestos separator from samples 5 and 6 (expected to be good material), with the performance when asbestos from sample 11 is used for the separator. In addition, the performance of a cell using zircar separator and containing the same amount of electrolyte should be examined to give an indication of performance when essentially no soluble inorganic components are present. At present a nickel hydrogen test cell using asbestos from samples 5 and 6 has performed well over about 50 cycles at 100% DOD, when activated with the normal quantity of KOH electrolyte. Similar comparative performance testing is planned for the other two test cells described above.

### Conclusions

Following the production of a nickel hydrogen cell lot that gave quite poor performance during acceptance testing, a strip of asbestos was obtained from the roll used as separator in this cell lot. Tests reported herein on this asbestos material indicate that about half the width of this asbestos roll (in this specific location along its length) contains significantly higher

quantities of a magnesium compound that exchanges hydroxide with KOH electrolyte, when compared with the other half of the roll width. Tests are indicated here that can be used to screen asbestos for such contamination prior to cell fabrication. It is possible that such reactions of the electrolyte with the asbestos could severely degrade cell performance. At present nickel hydrogen cell tests are underway using separator from each half of this roll to determine how cell performance correlates with the levels of contaminants observed.

# Figure 1. Acid Analysis— Virgin Asbestos Separator



# Figure 2. Acid Extraction

Weight Loss vs. Chelate Wt., A Samples

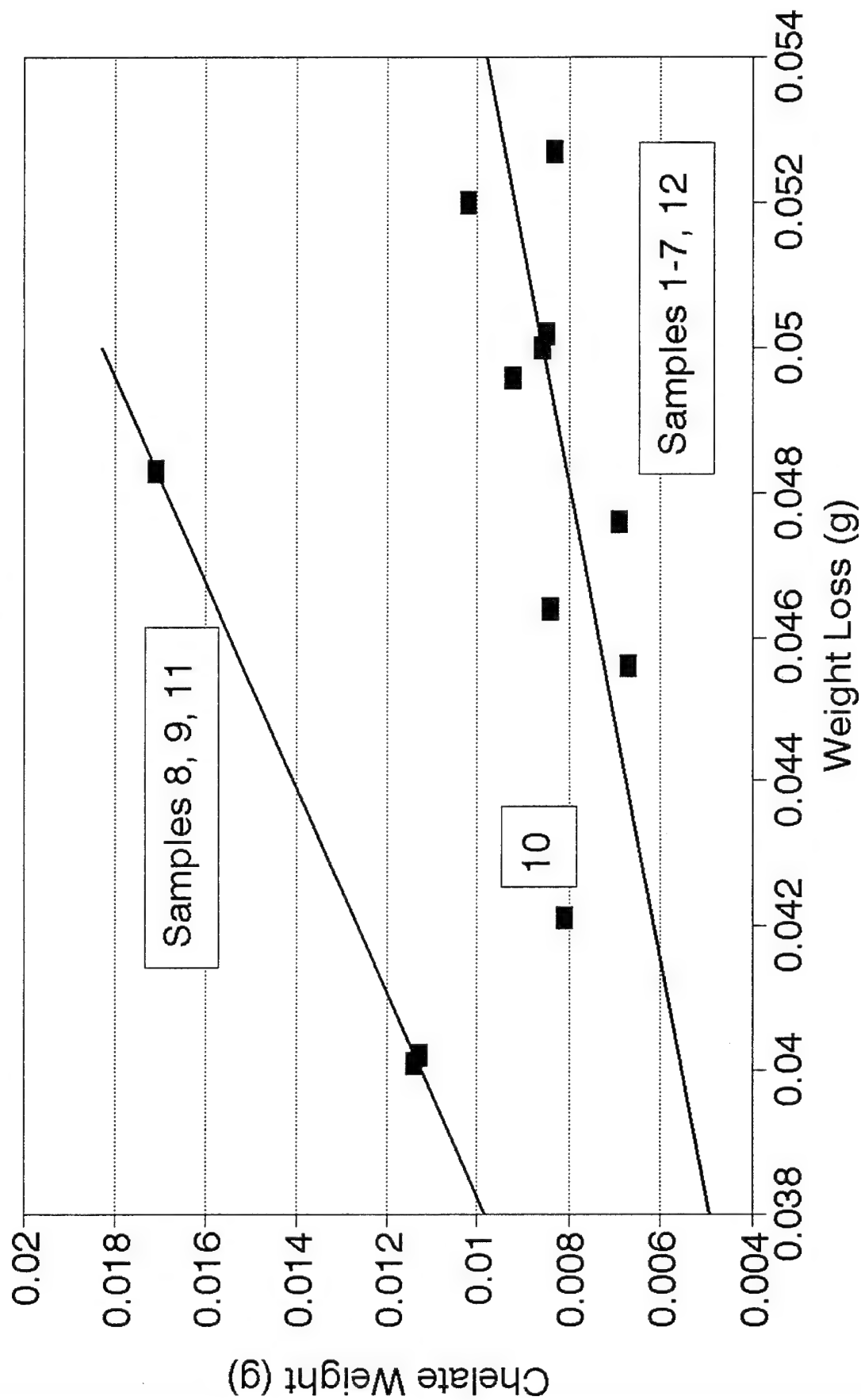
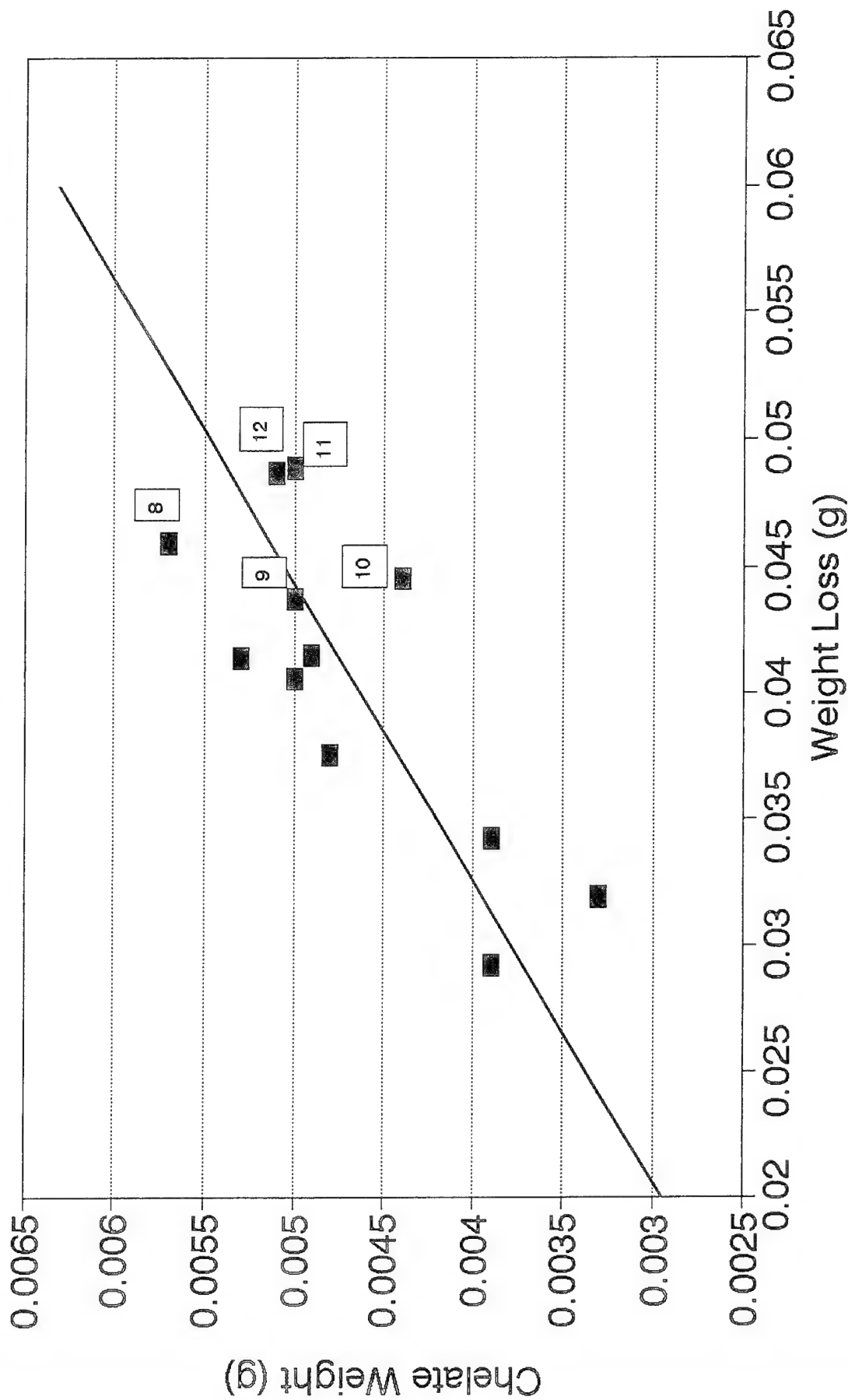
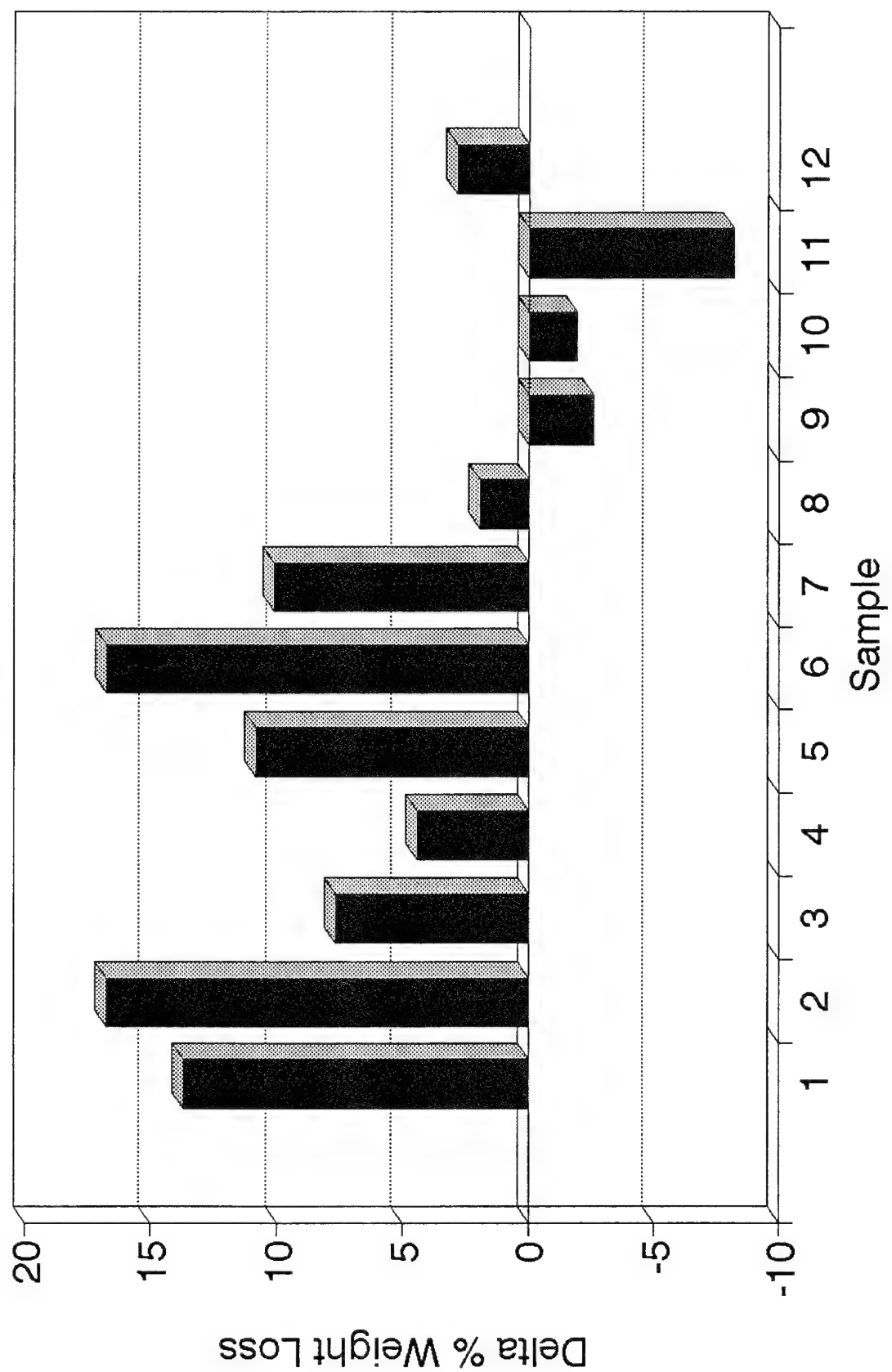


Figure 3. Acid Rinse After KOH Treat  
Weight Loss vs. Chelate Wt., B Samples



**Figure 4. Delta % Weight Loss**  
A % Loss - B % Loss During Acid Rinse





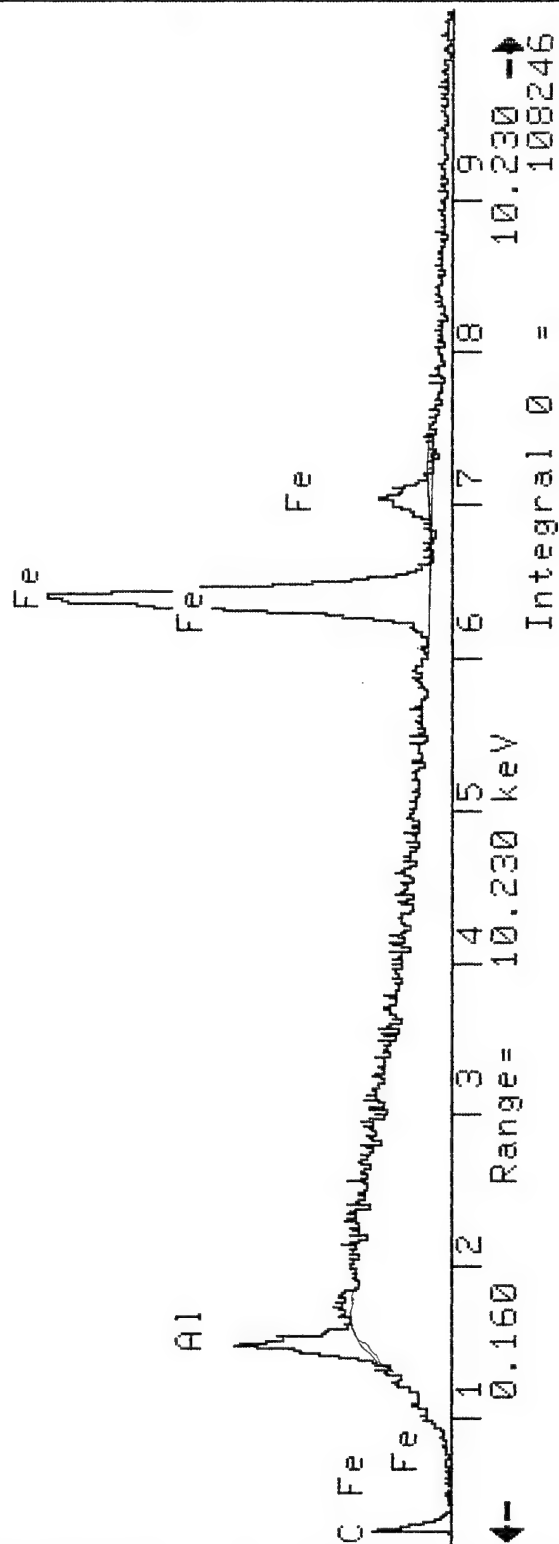
# **APPENDIX 1**

## **EDAX Spectra for Chelate Precipitates Obtained from Acid Rinse Solution for Samples 1A to 12A**

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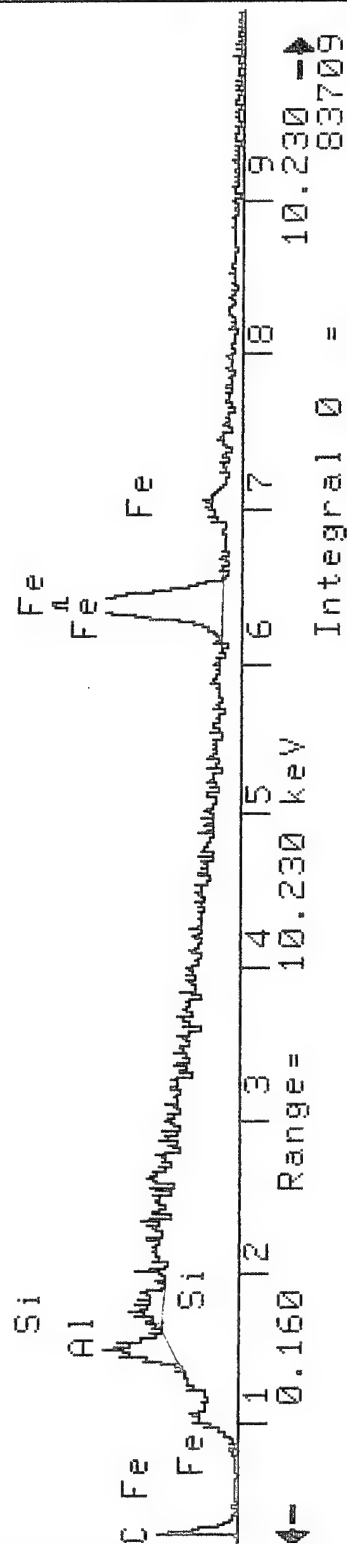
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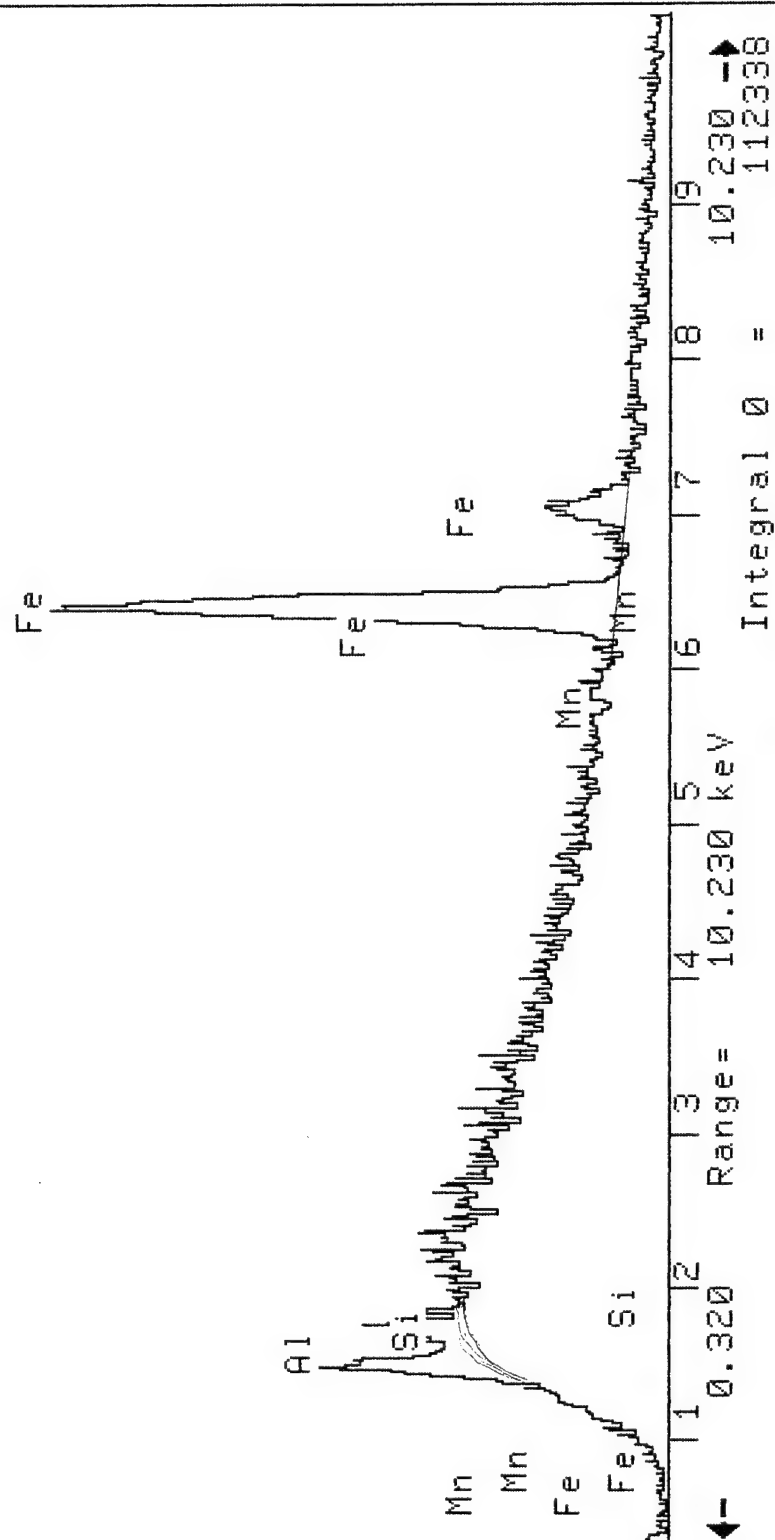
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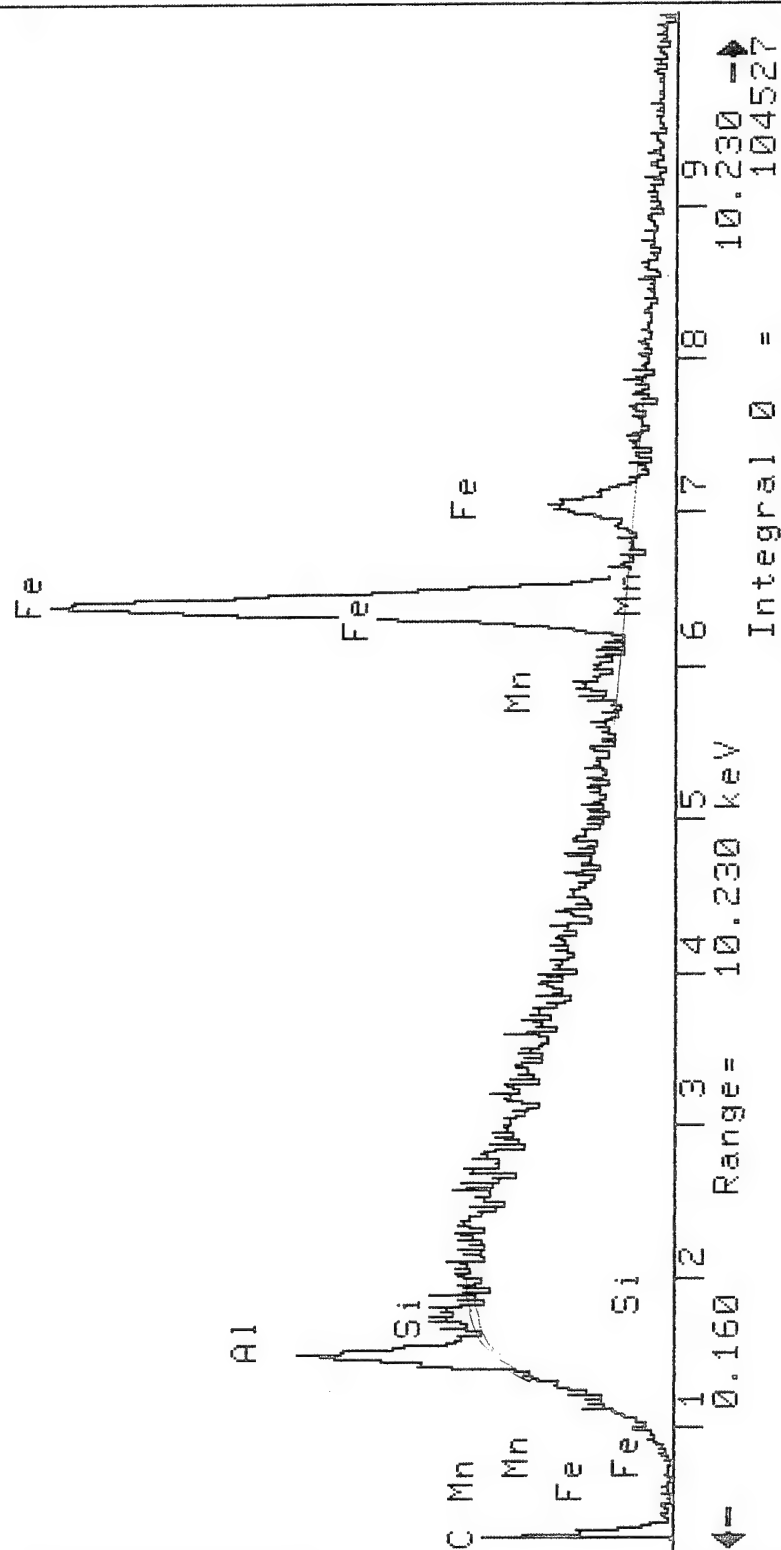
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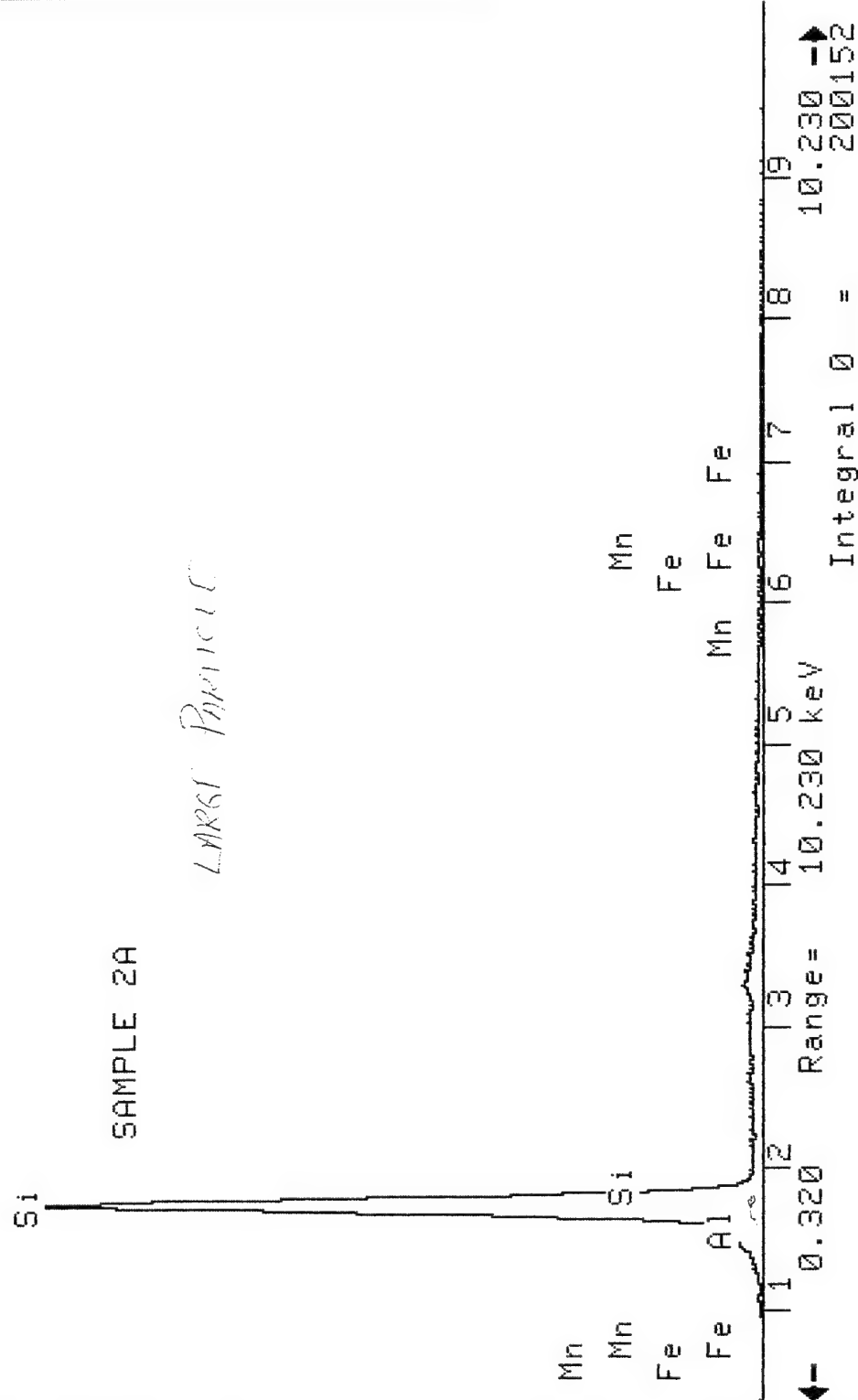
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SAMPLE 2A



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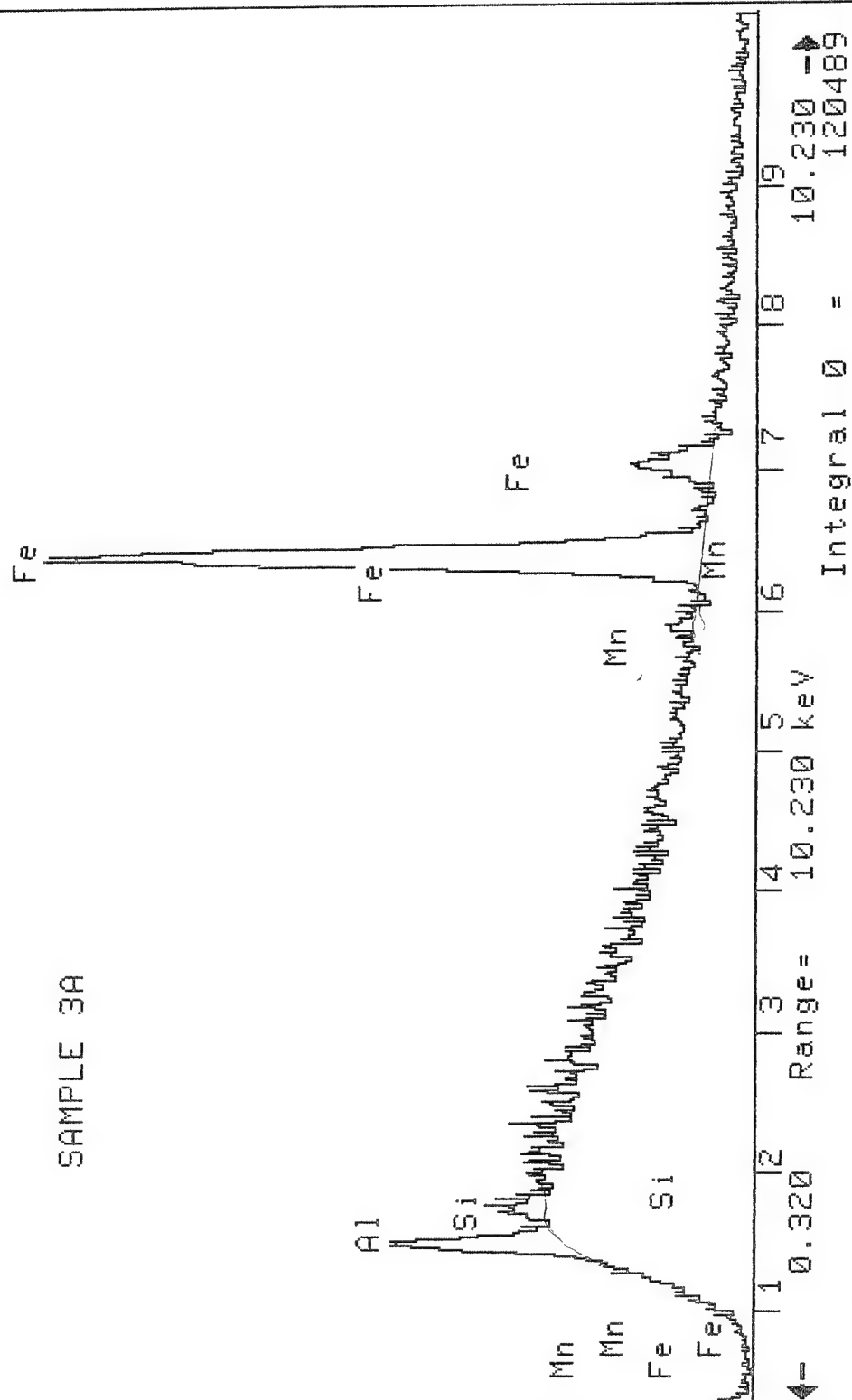
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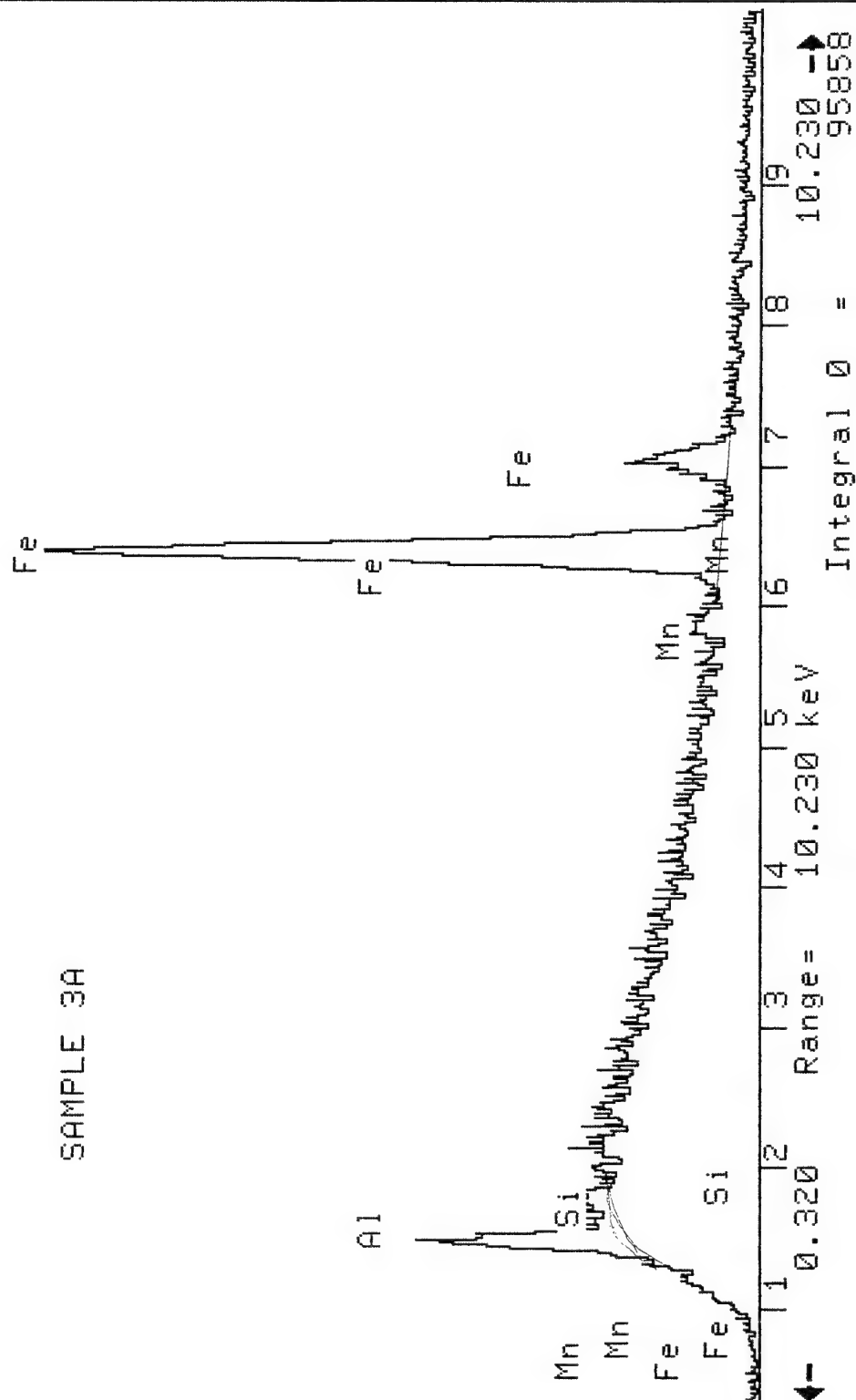




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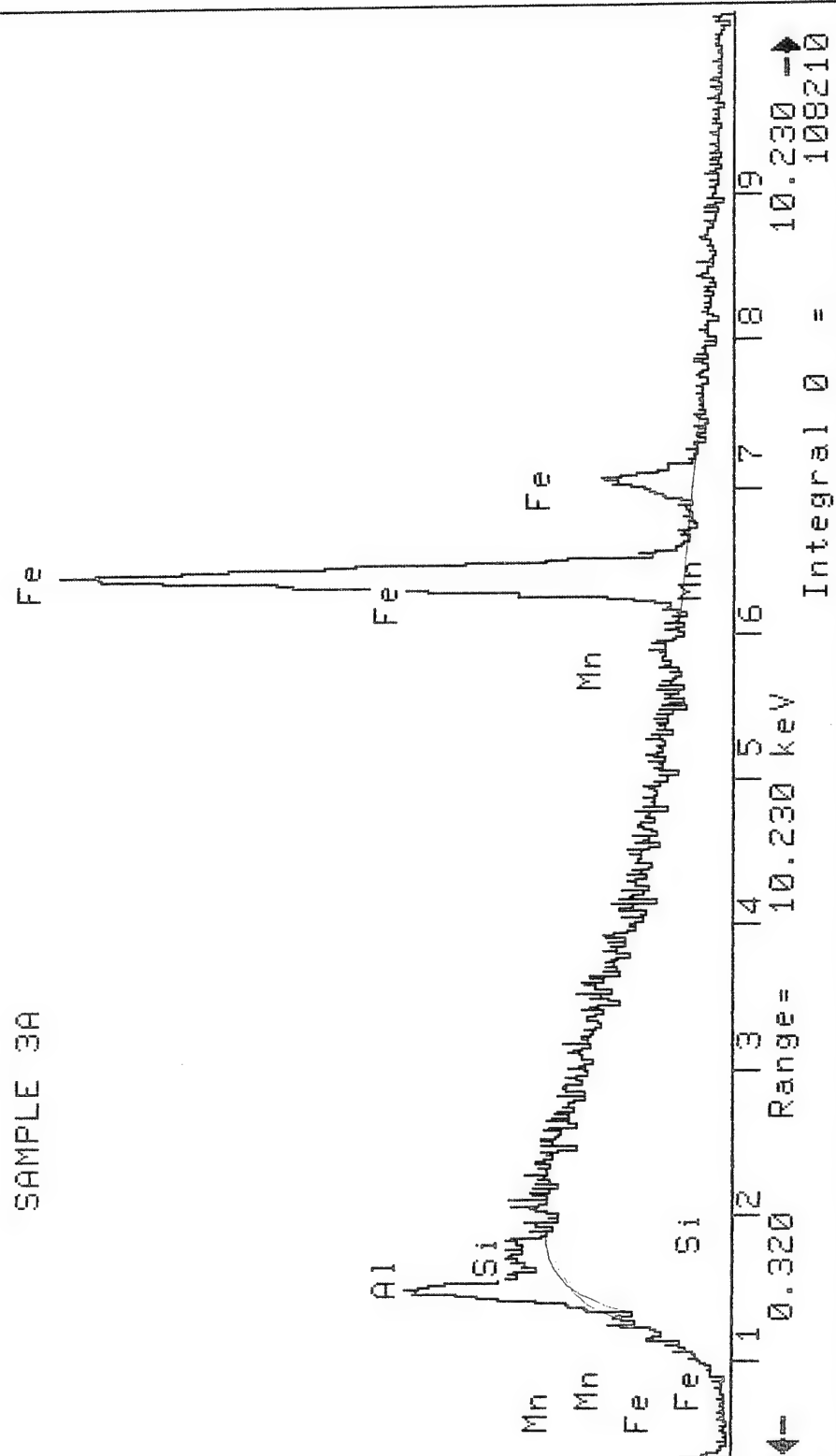
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SAMPLE 3A

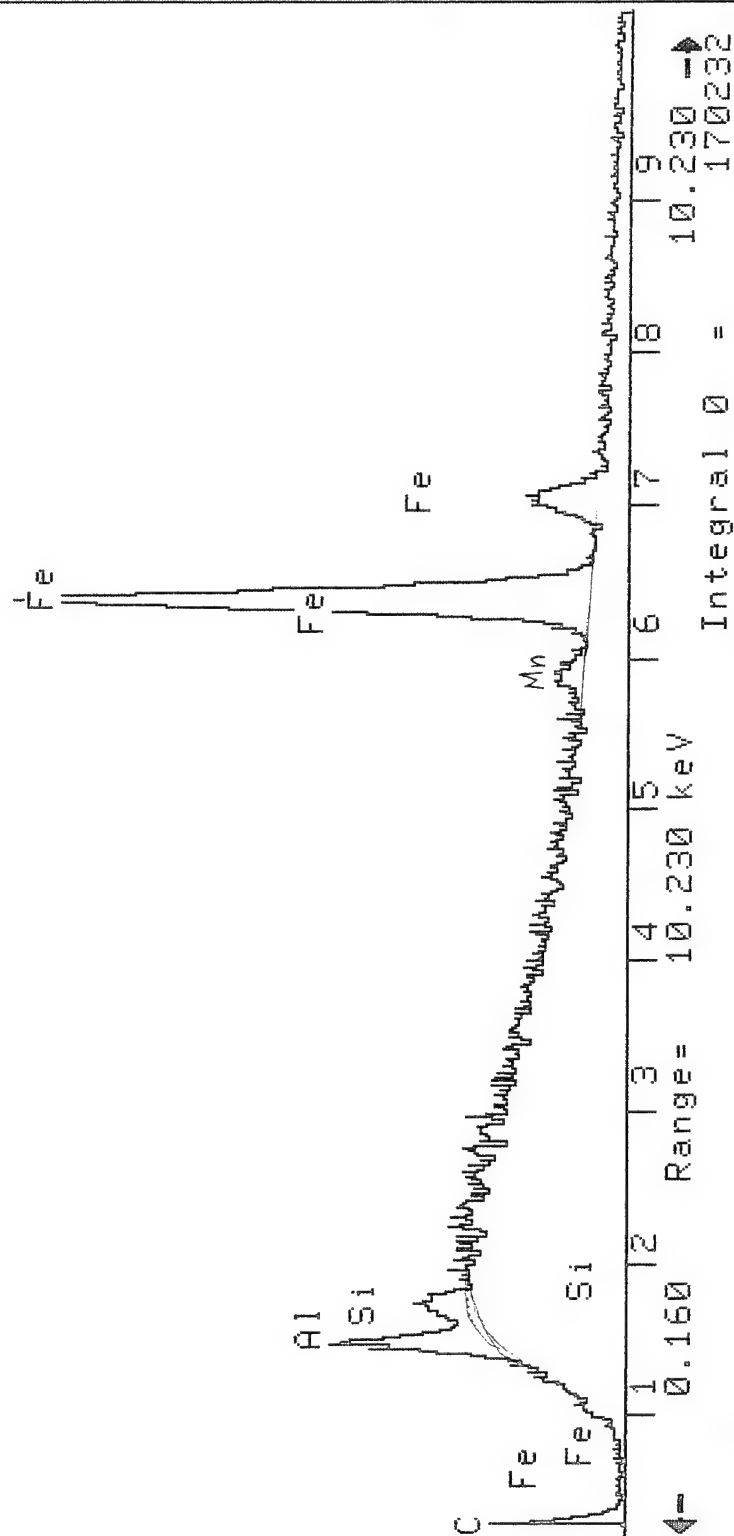




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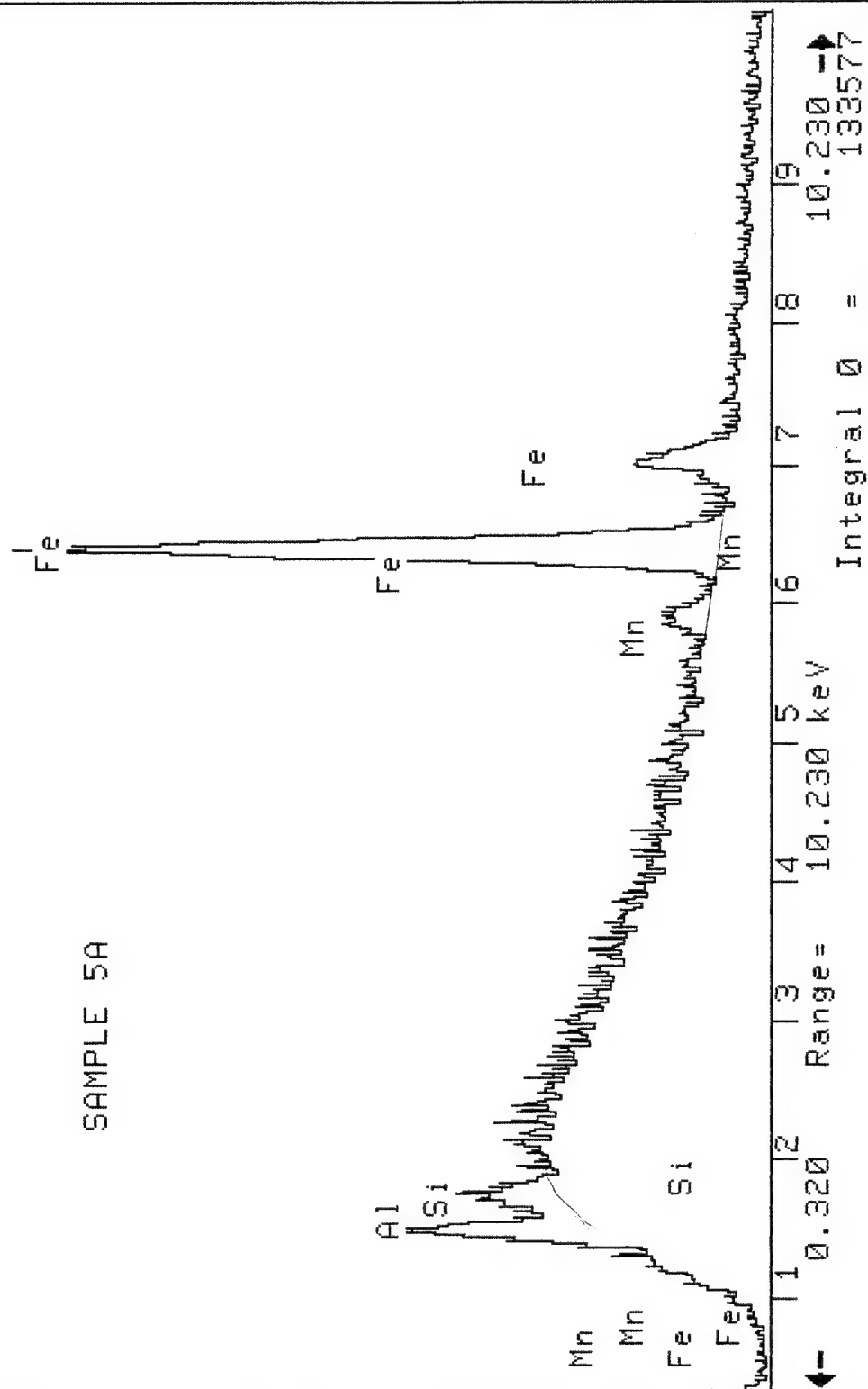
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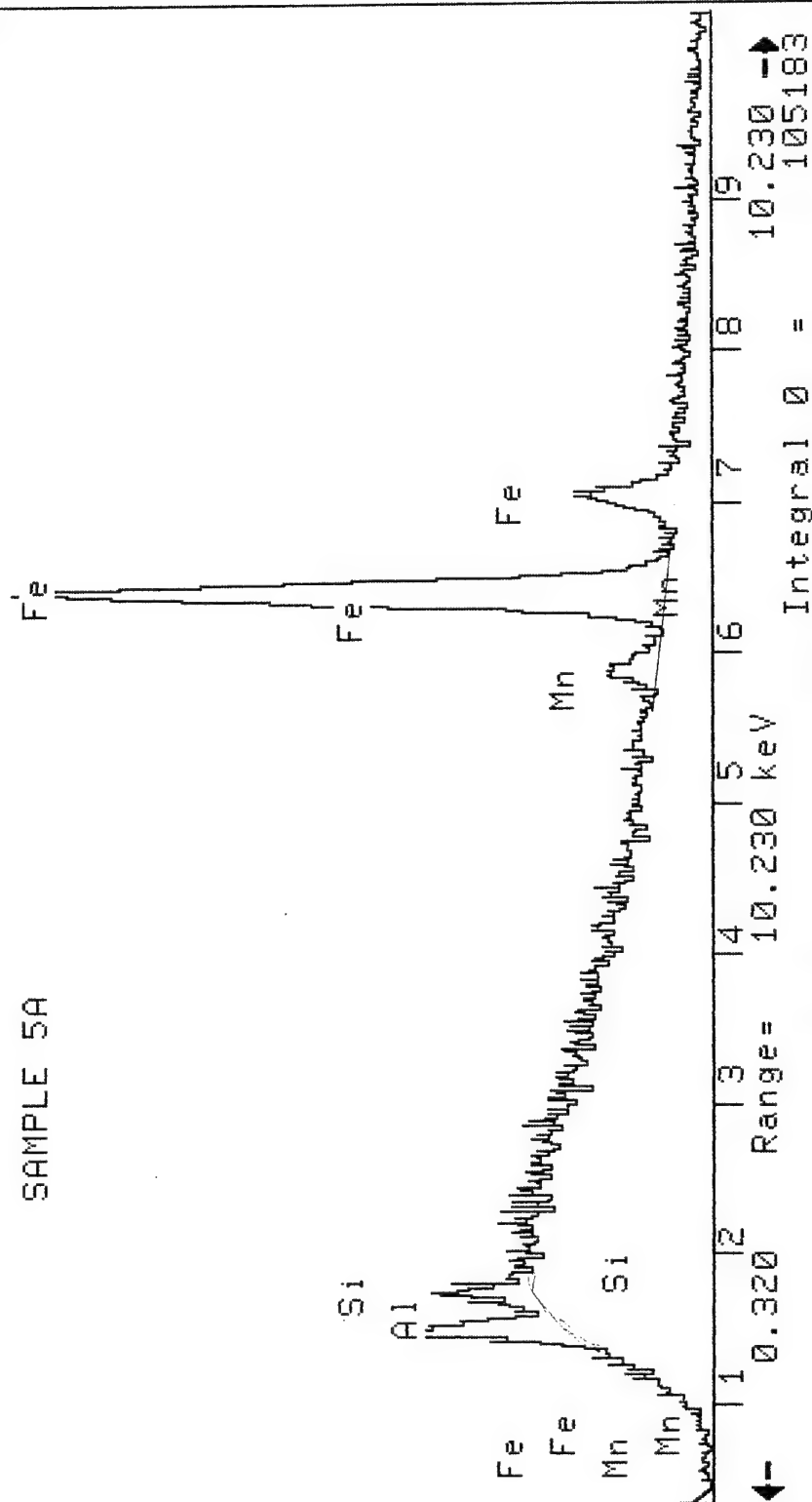




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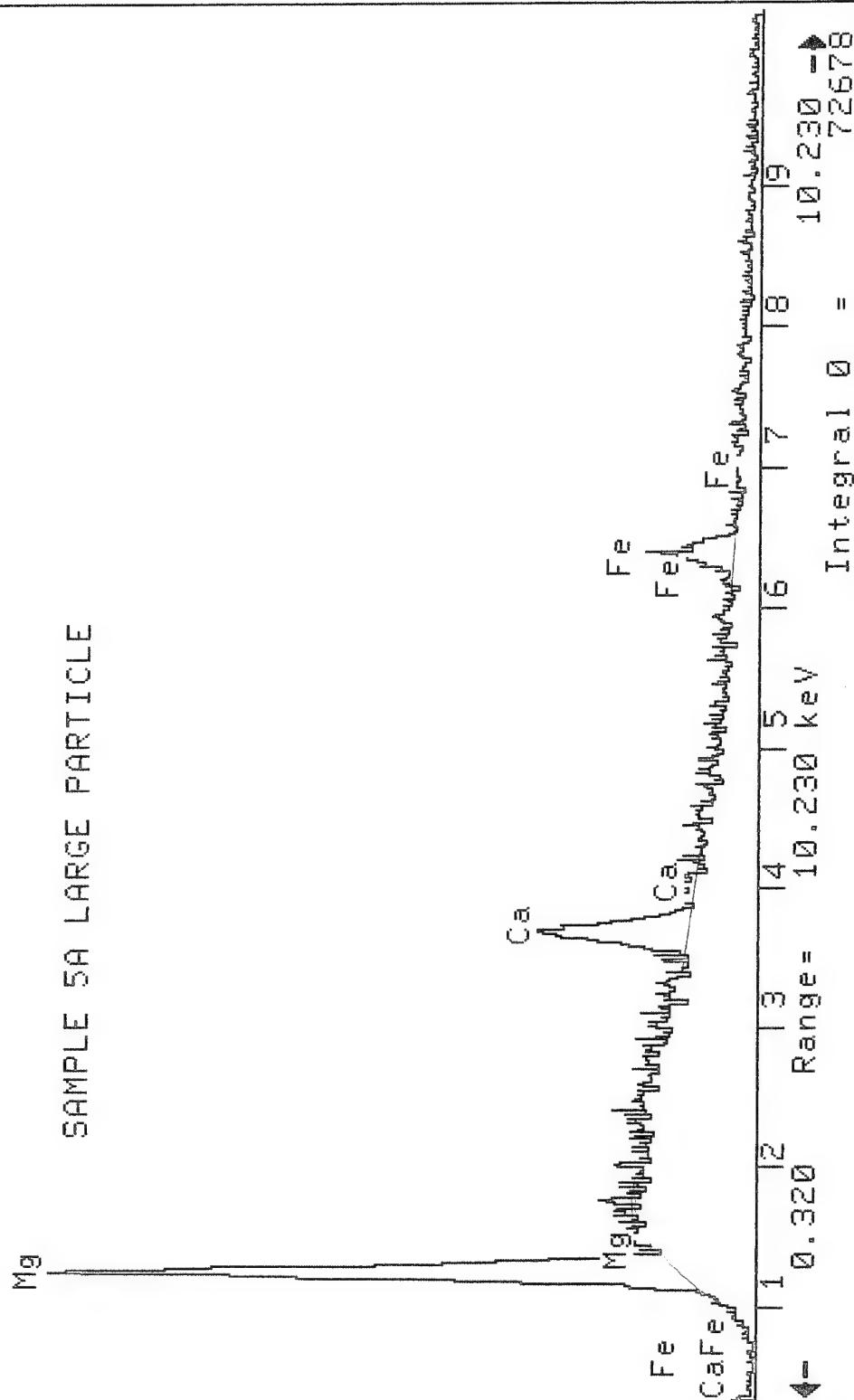
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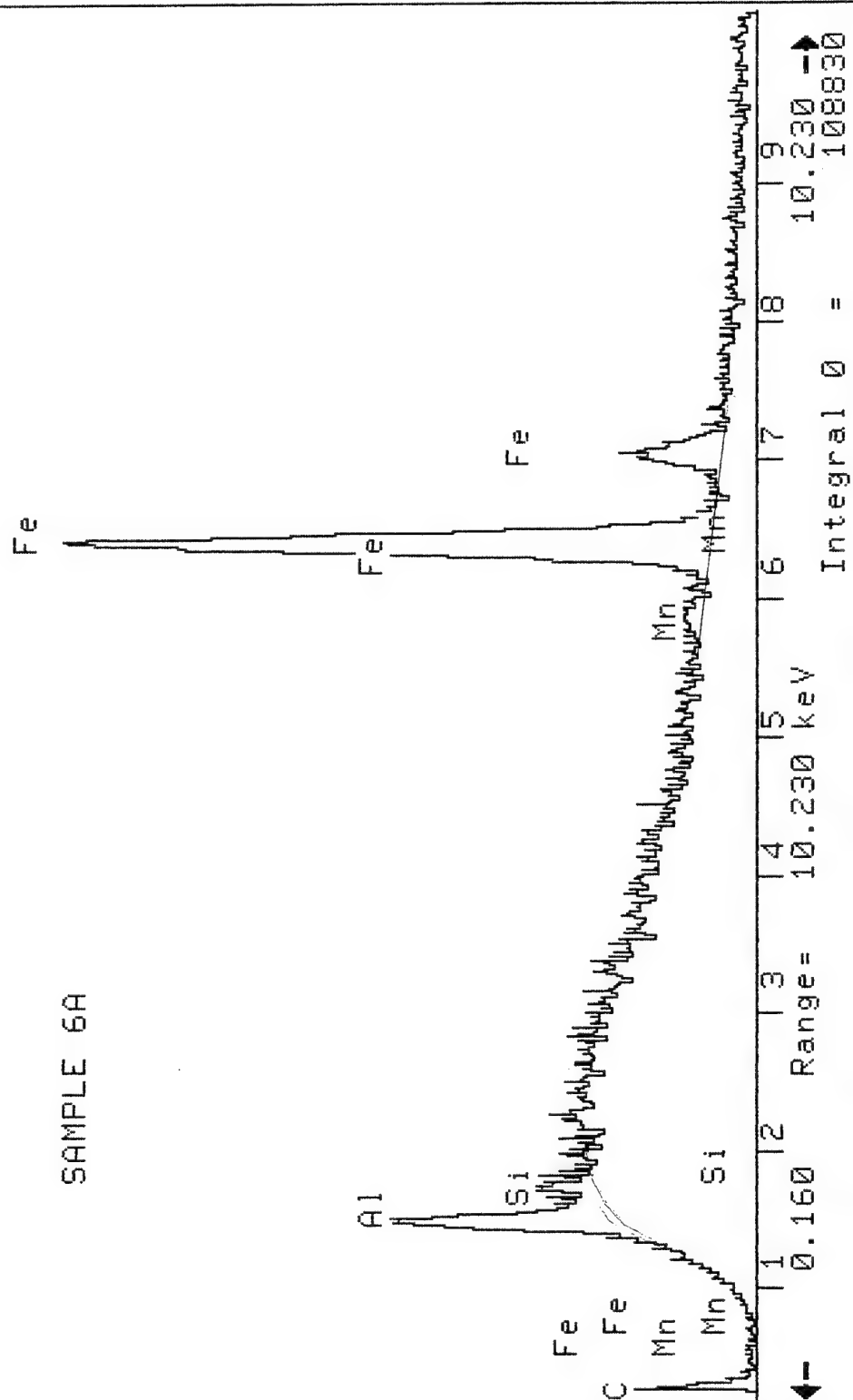
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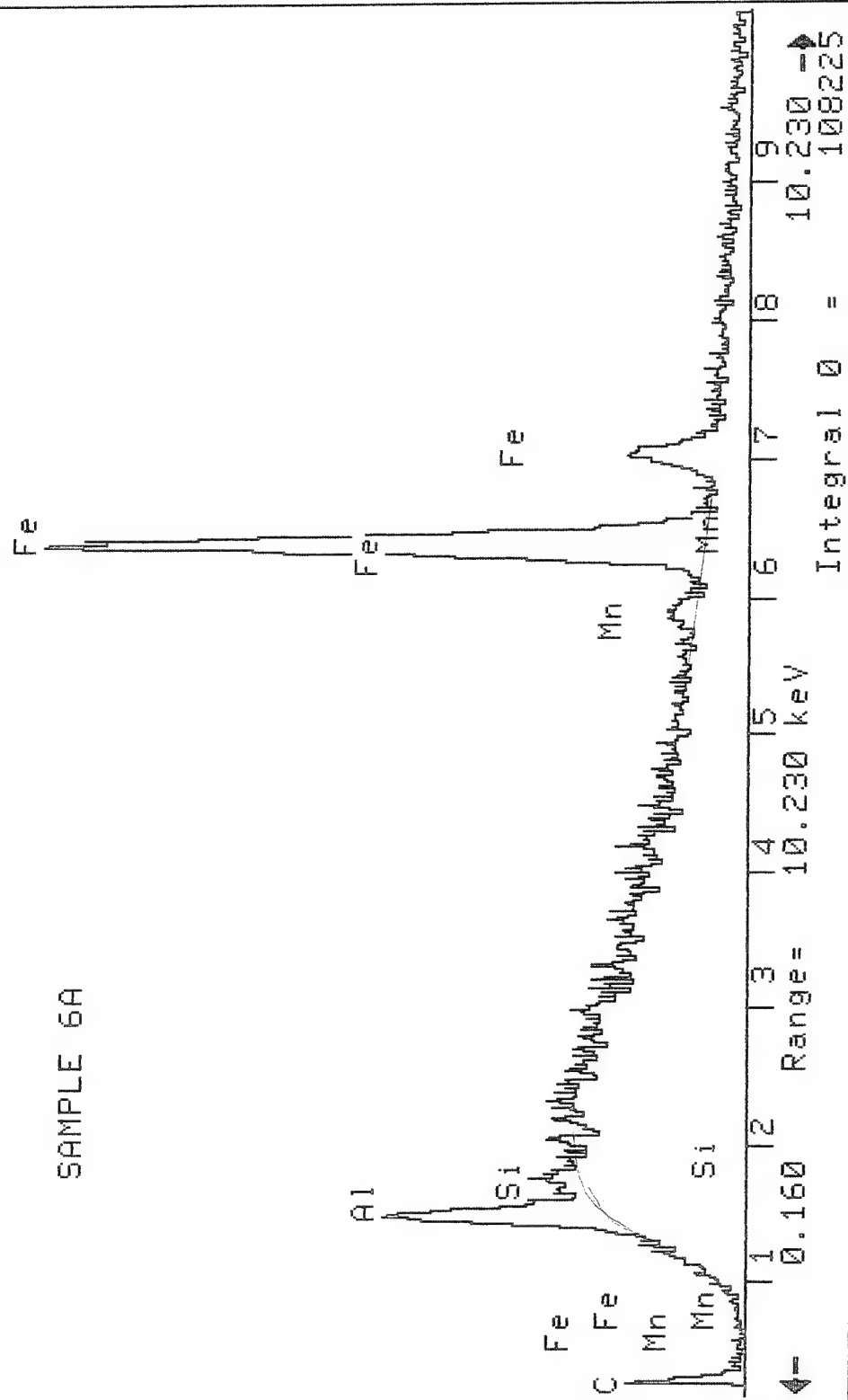
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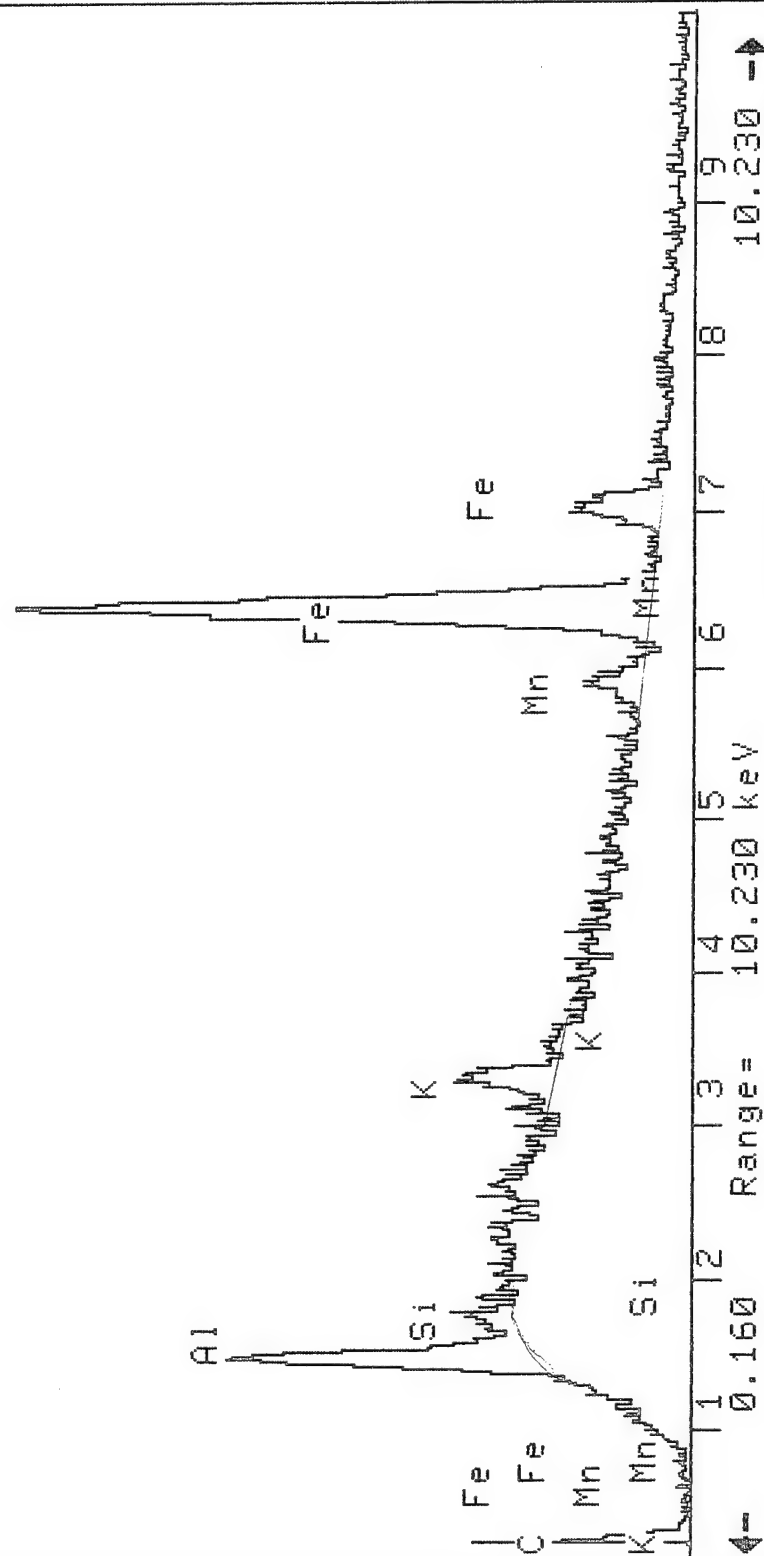
SAMPLE 6A





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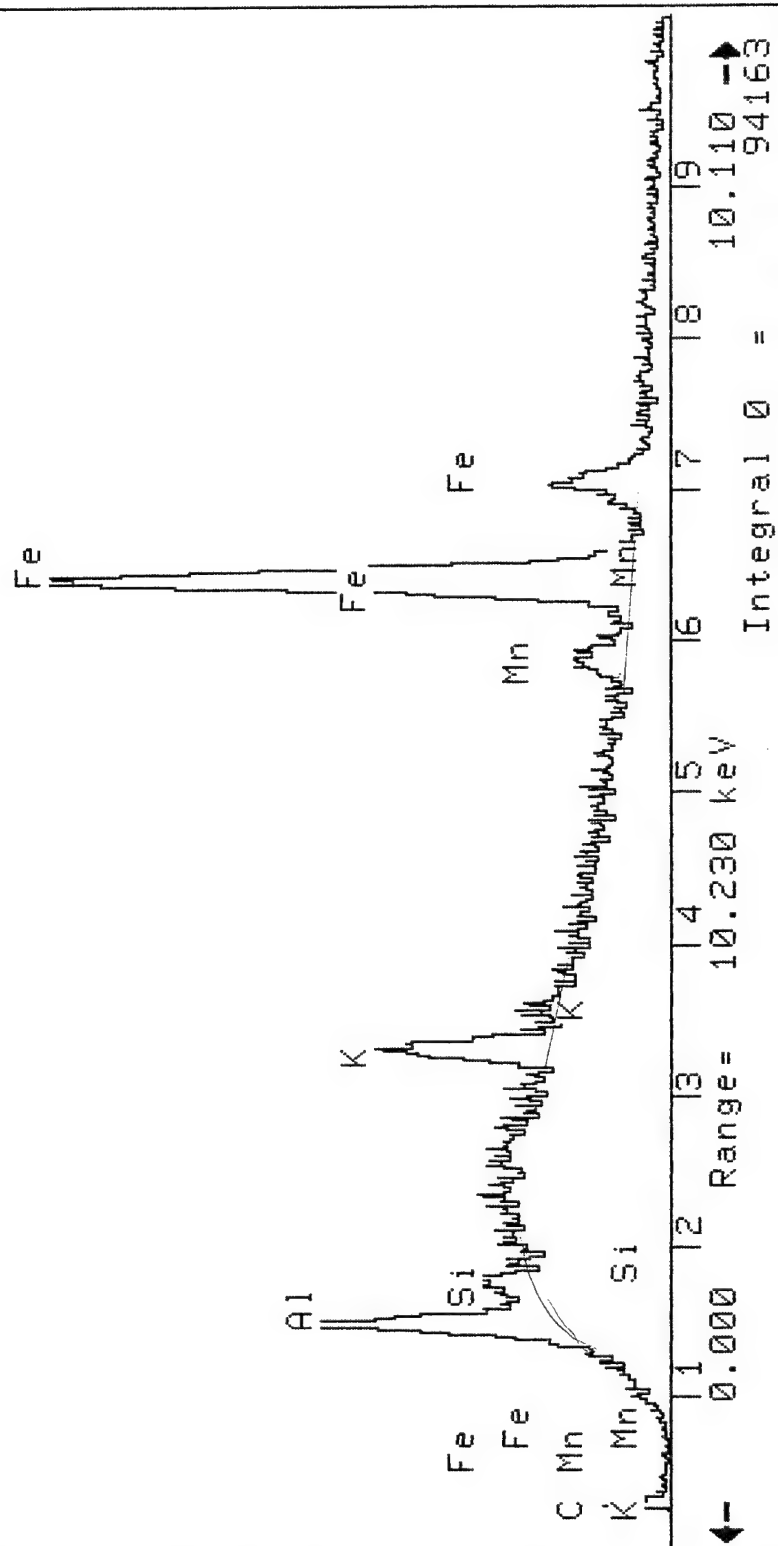


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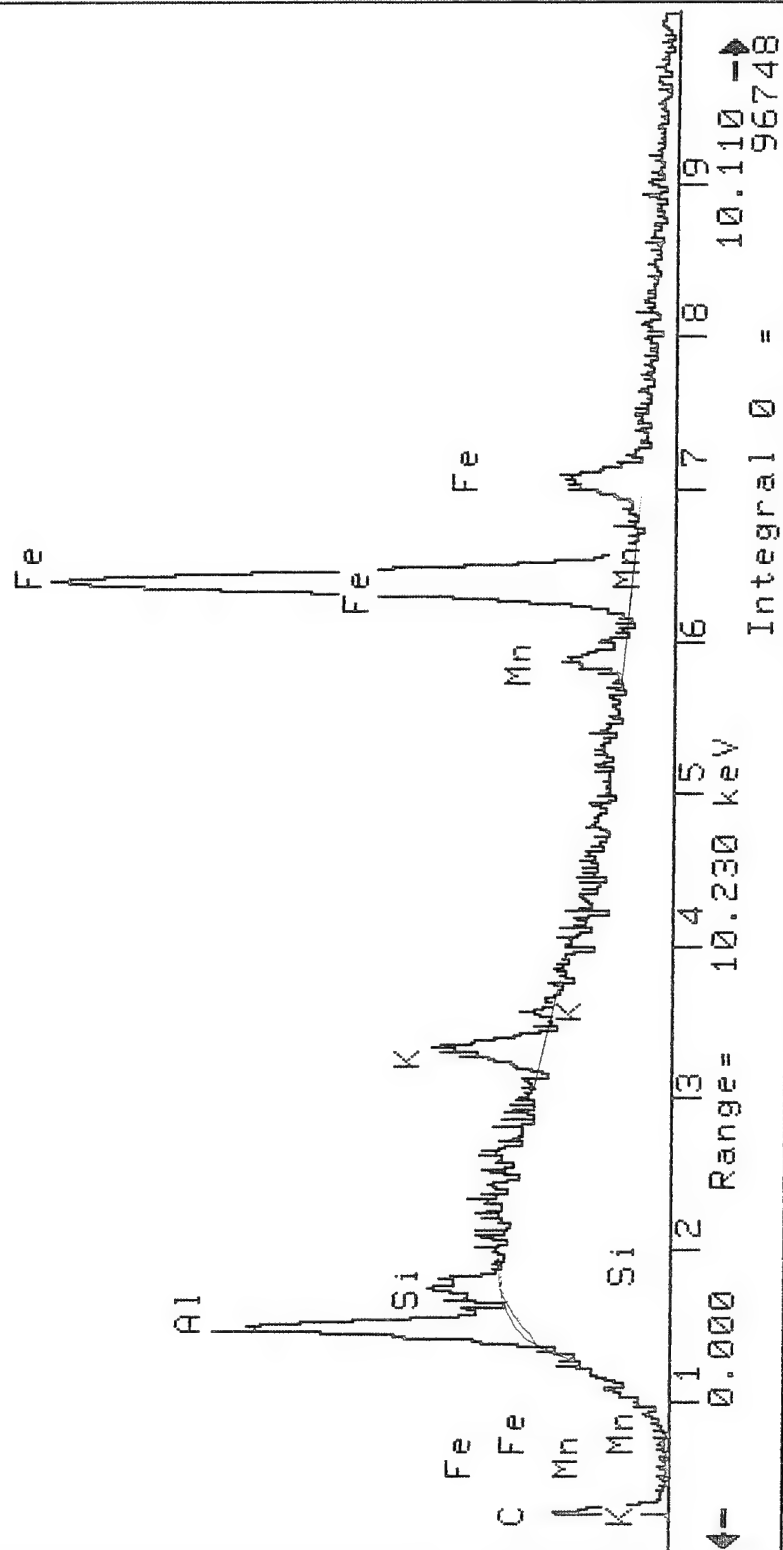
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SAMPLE 7A

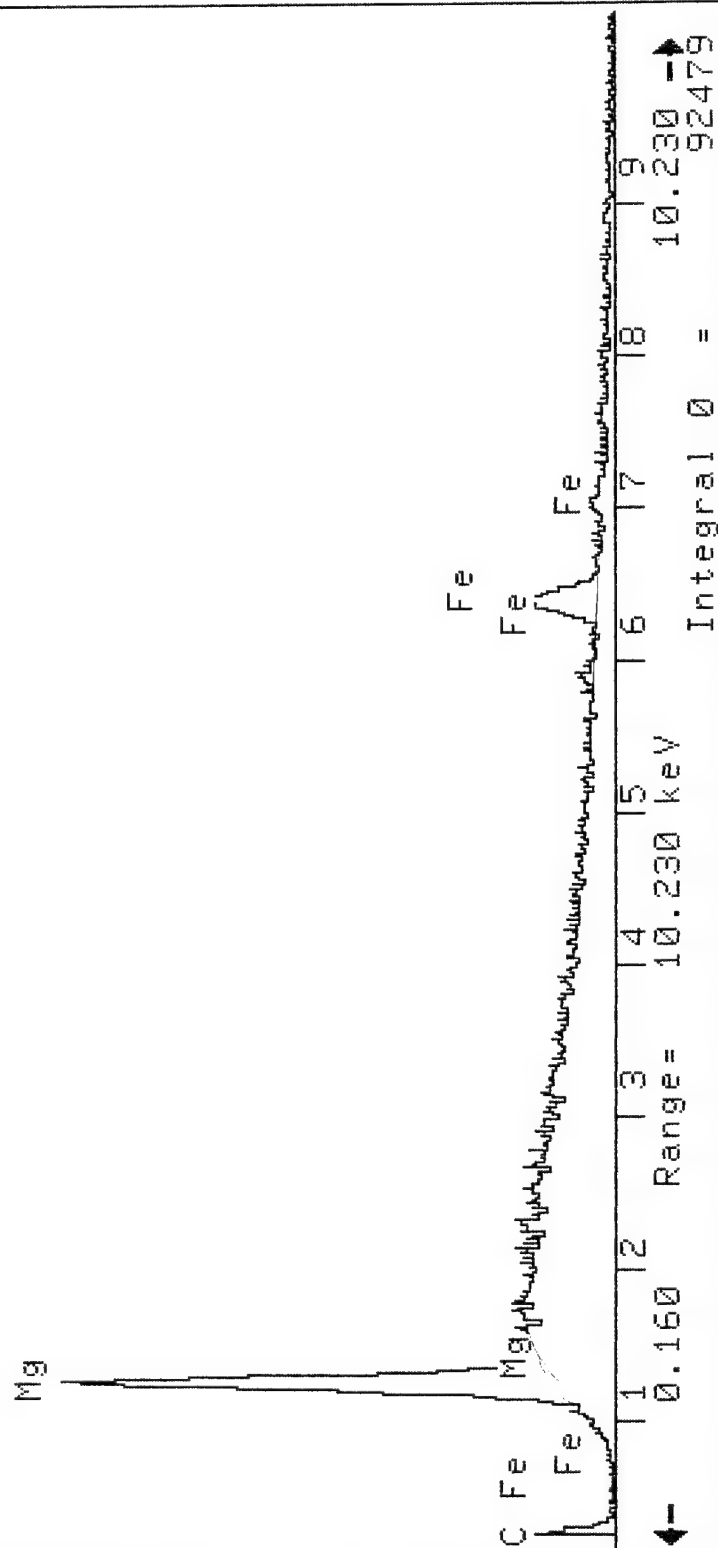




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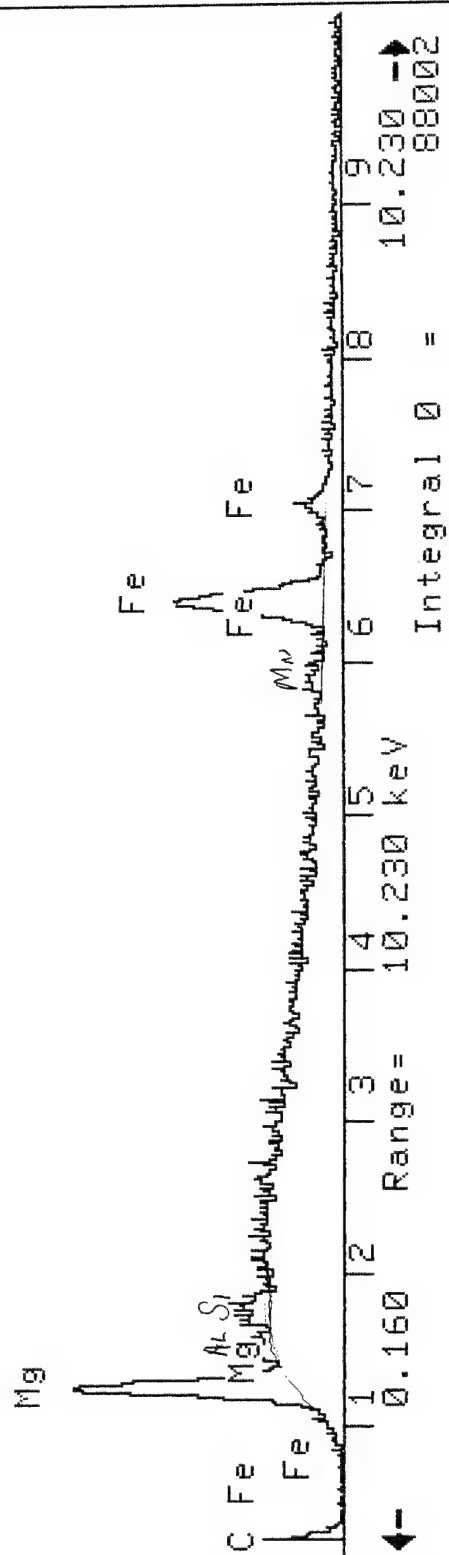
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Elapsed= 115 secs
```

EDS spectrum showing peaks for C, Fe, Mo, Al, Si, and Fe. The x-axis is labeled 'Range = 10.230 keV' and 'Integral 0 = 97269'. The y-axis is labeled 'Fe'.

8-Oct-1992 15:09:43

Vert= 2000 counts Disp= 1 Preset= Off Elapsed= 105 secs

SAMPLE 9A

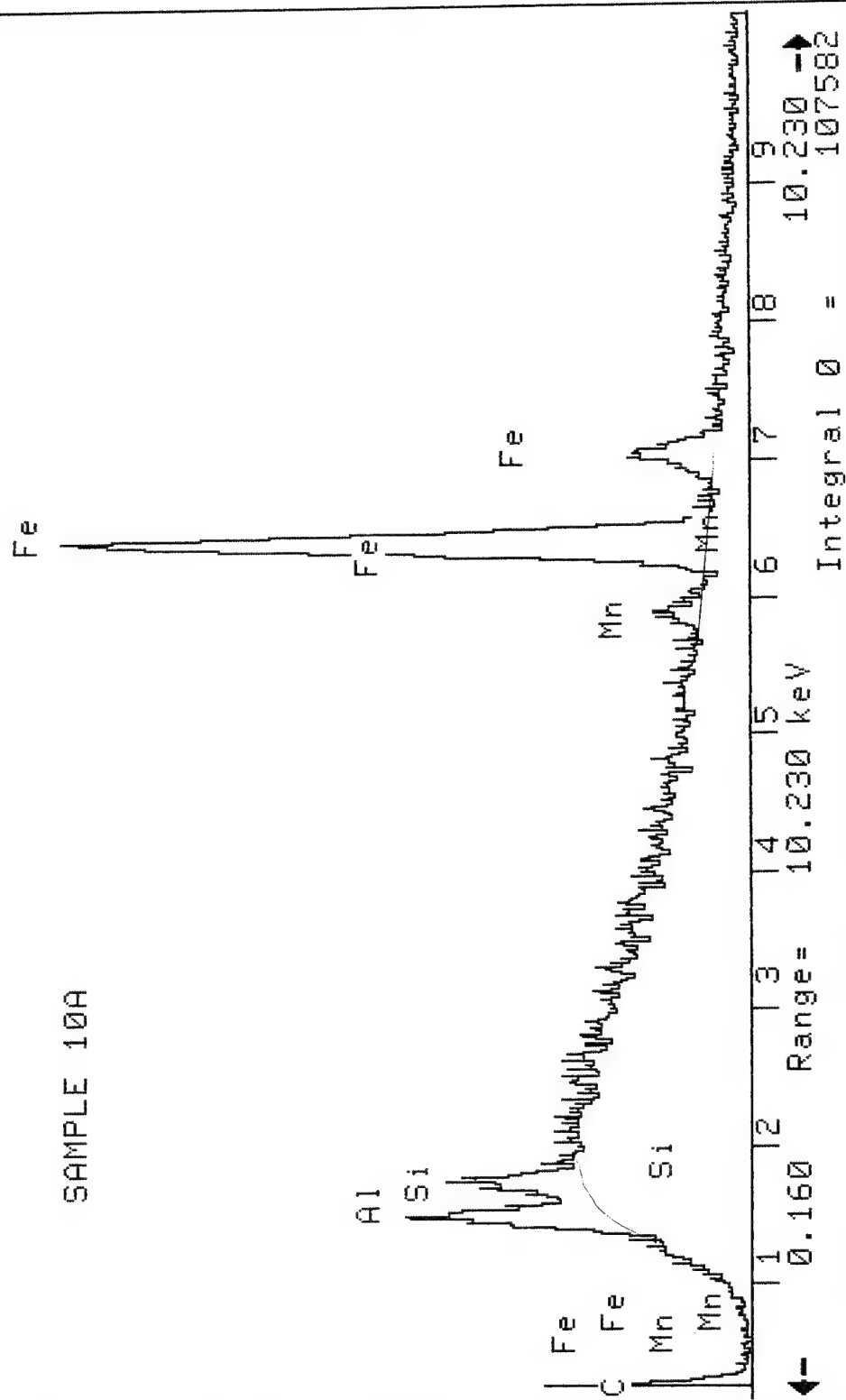




14-Oct-1992 10:18:40

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 50 secs

SAMPLE 10A

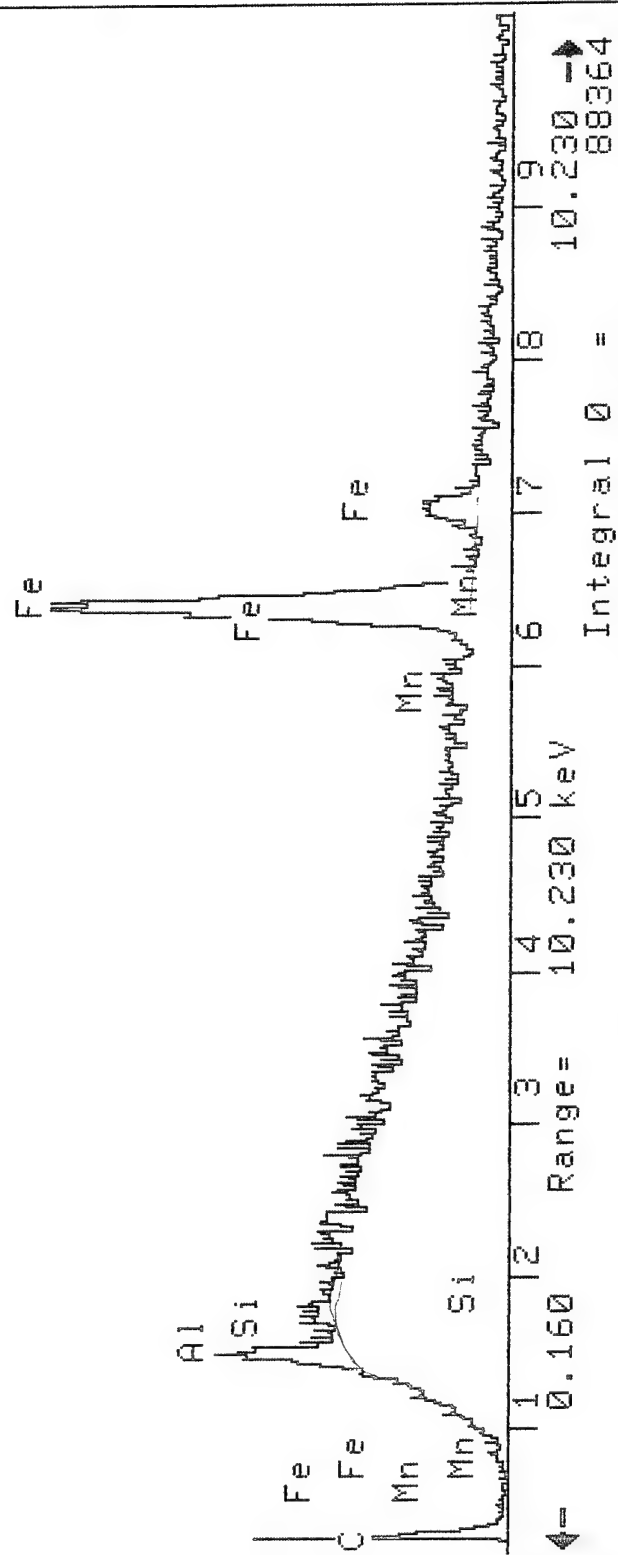


14-Oct-1992 10:48:08

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 51 secs

SAMPLE 10A

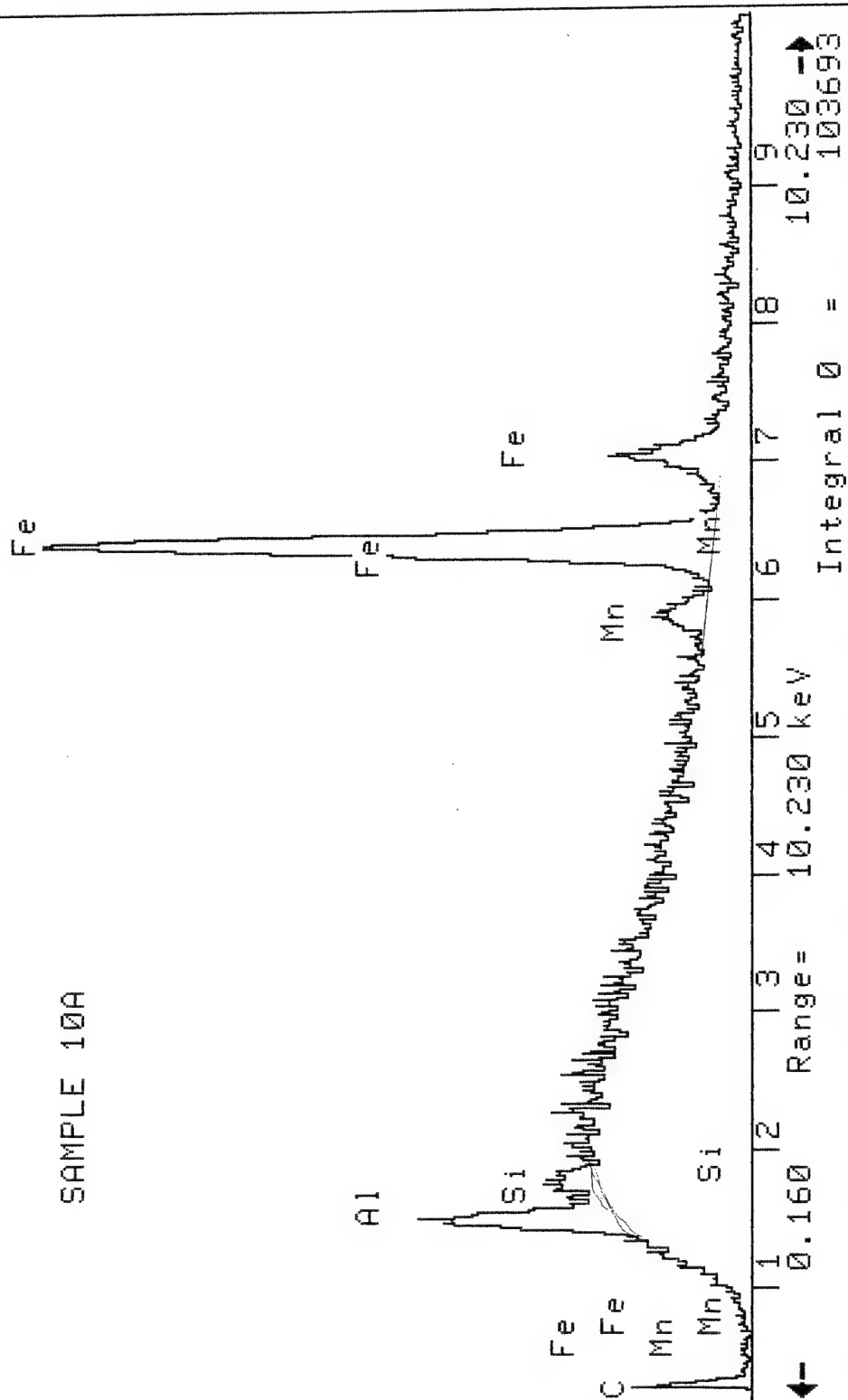
42



14-Oct-1992 12:44:40

Vert = 1000 counts    Disp = 1    Preset = Off    Elapsed = 45 secs

SAMPLE 10A

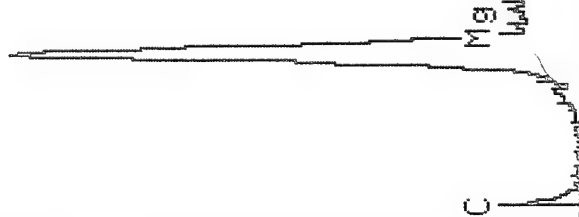


14-Oct-1992 10:10:31

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 20 secs

SAMPLE 10A LARGE PARTICLE  
*SAW MANY  
(300 SHAPES)*

Mg



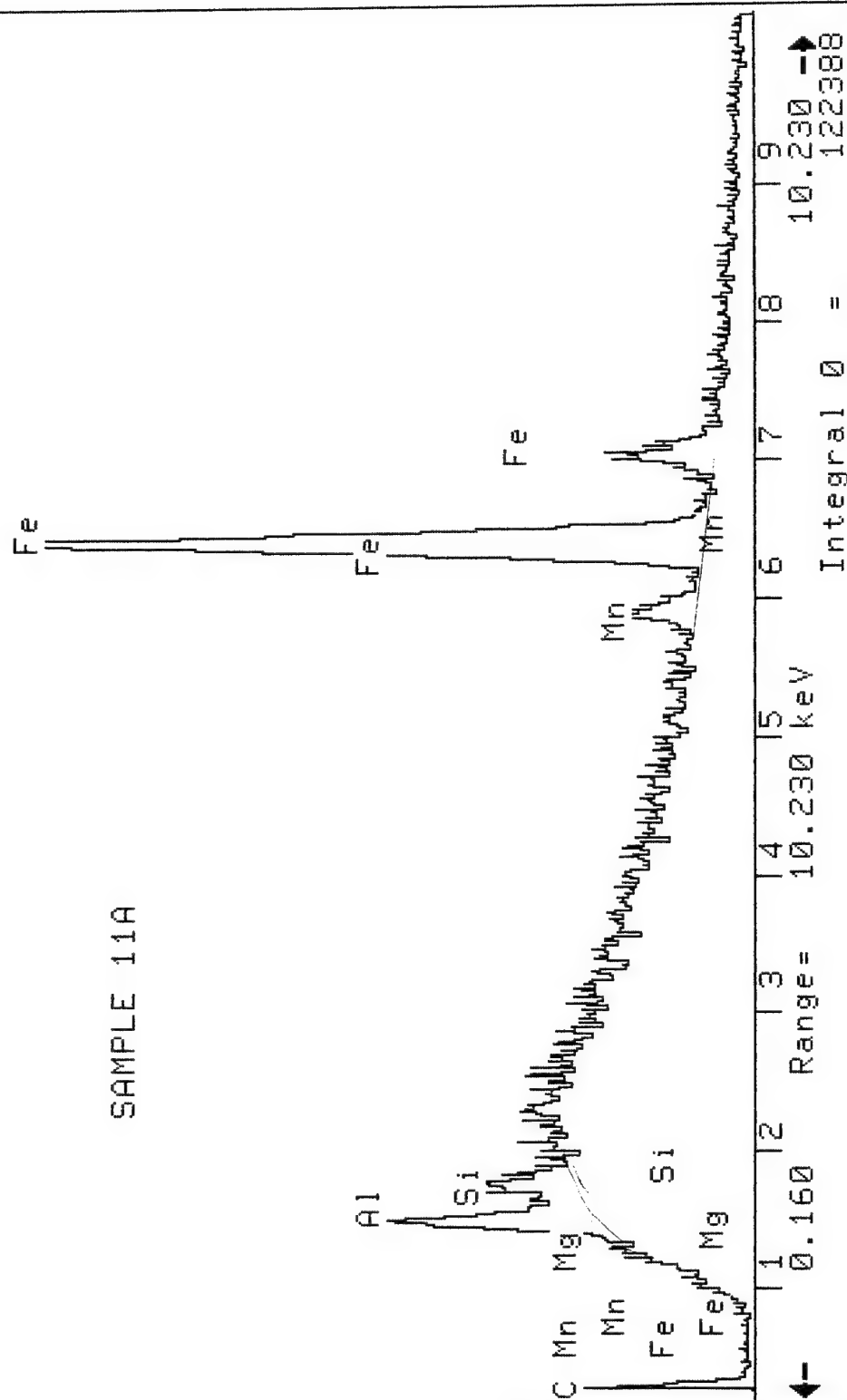
4- 0.160 Range= 10.230 keV Integral 0 = 40605



14-Oct-1992 13:27:54

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 63 secs

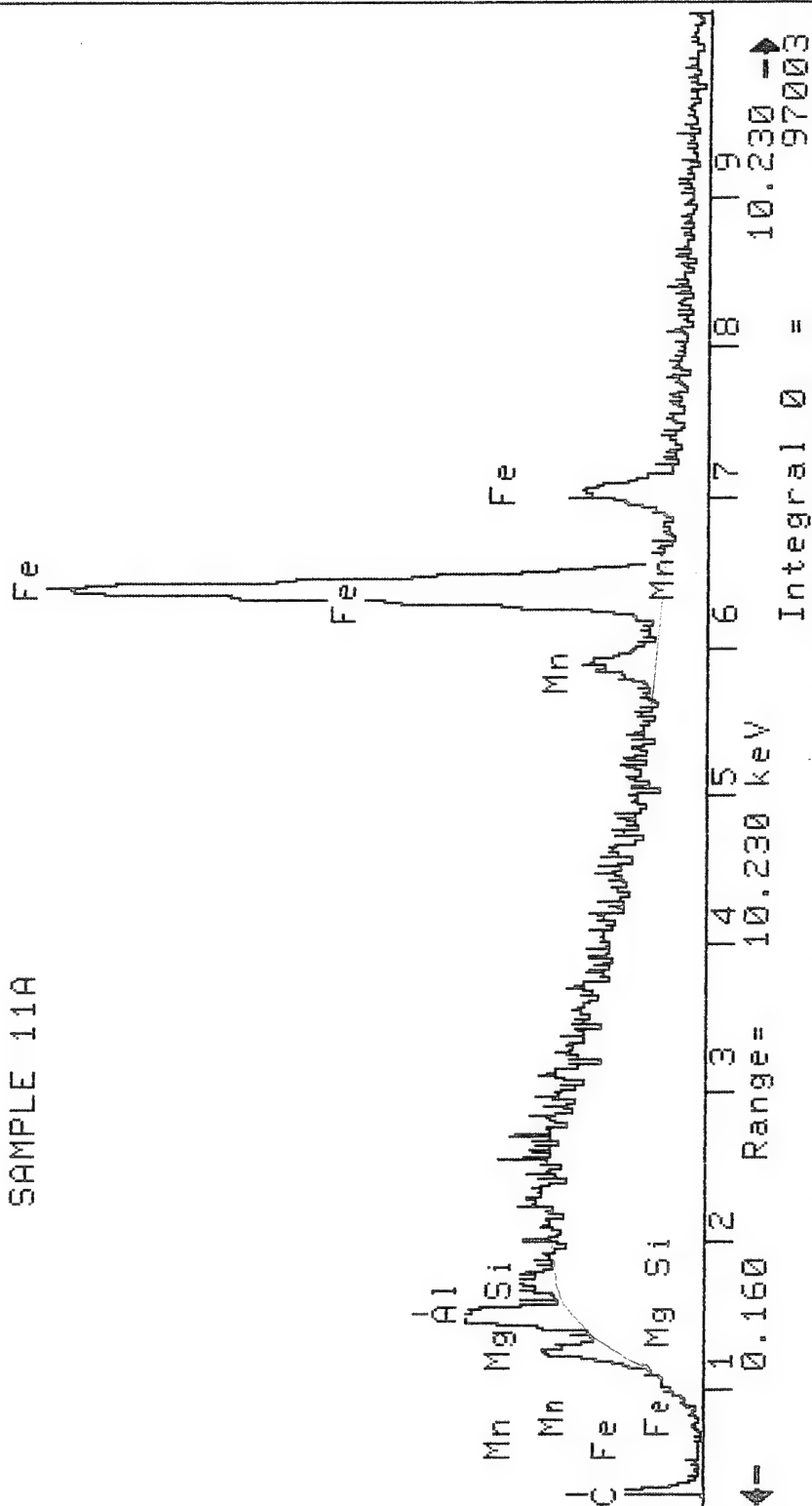
SAMPLE 11A



14-Oct-1992 13:24:58

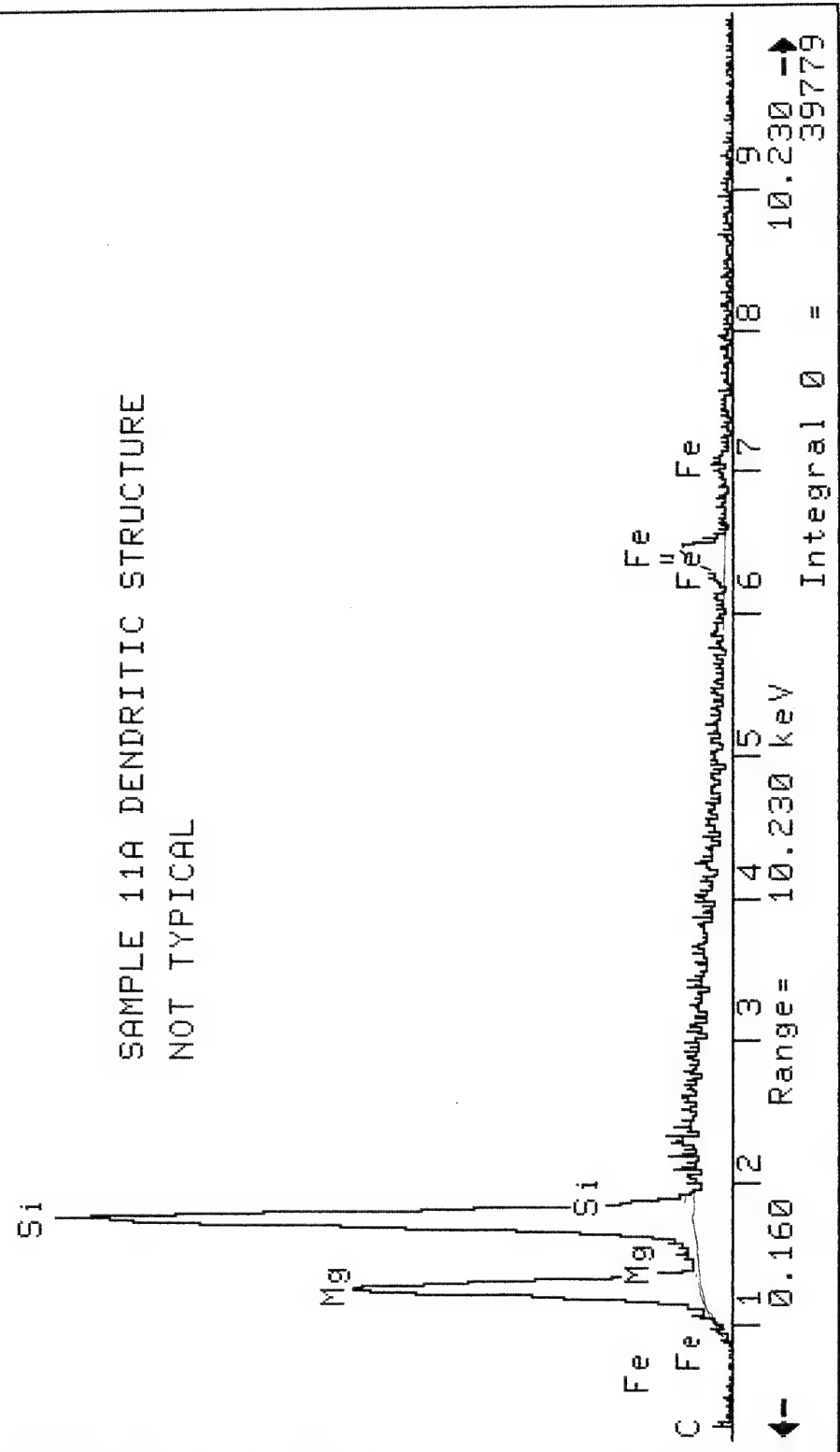
Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 51 secs

SAMPLE 11A



14-Oct-1992 13:18:12

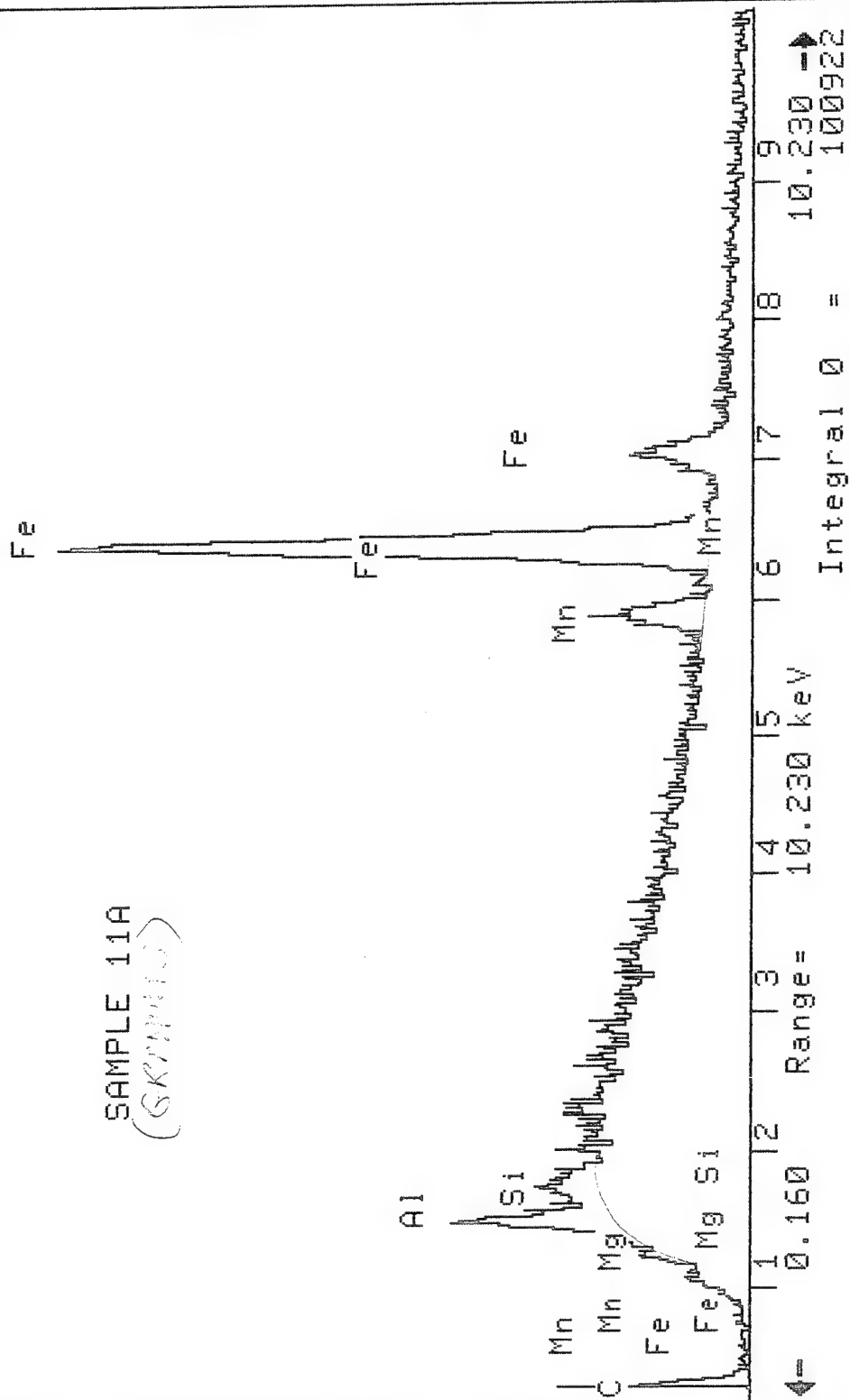
Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 13 secs



14-Oct-1992 13:30:32

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 50 secs

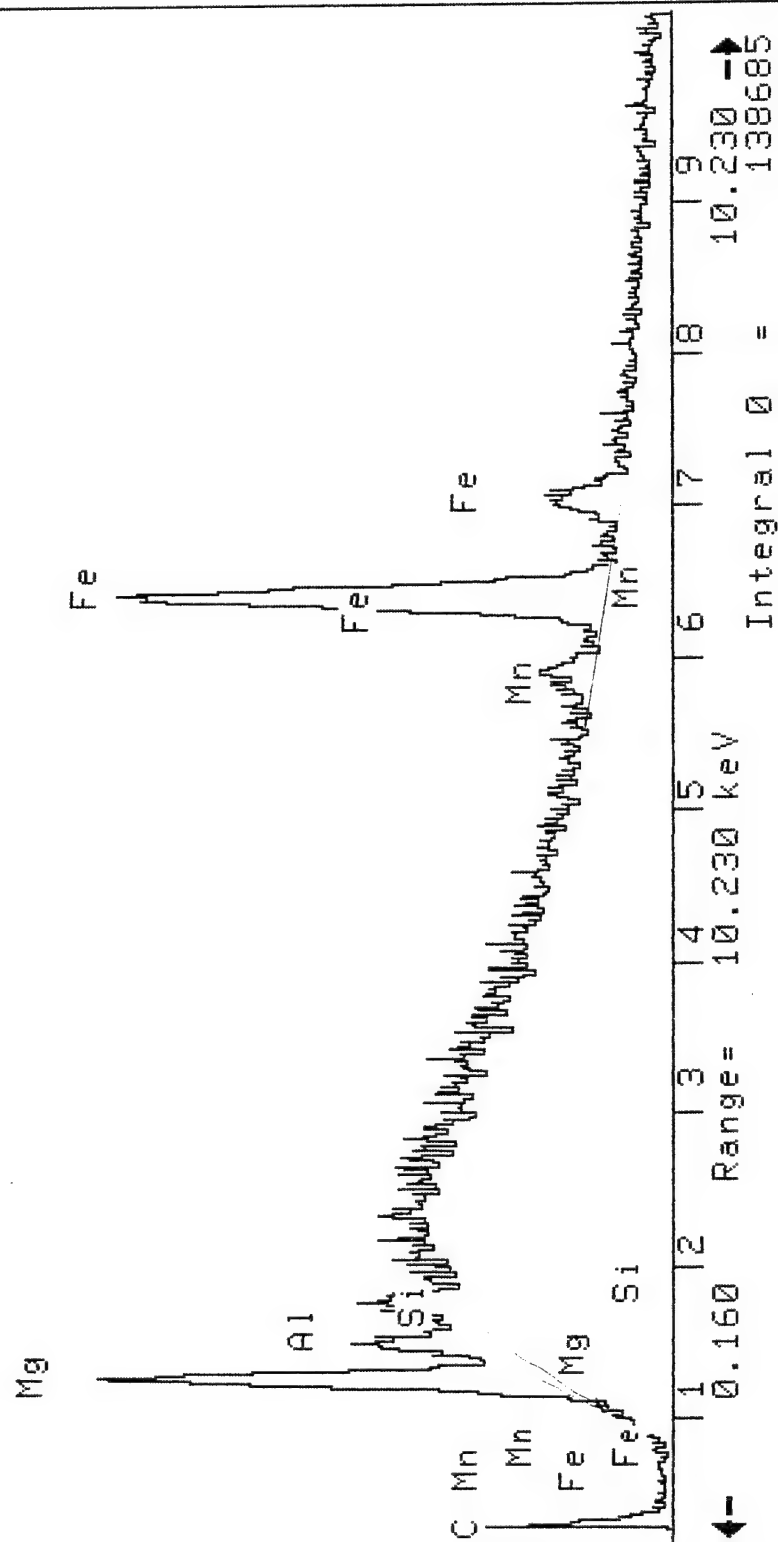
SAMPLE 11A  
(GRINDING)



14-Oct-1992 13:36:50

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 90 secs

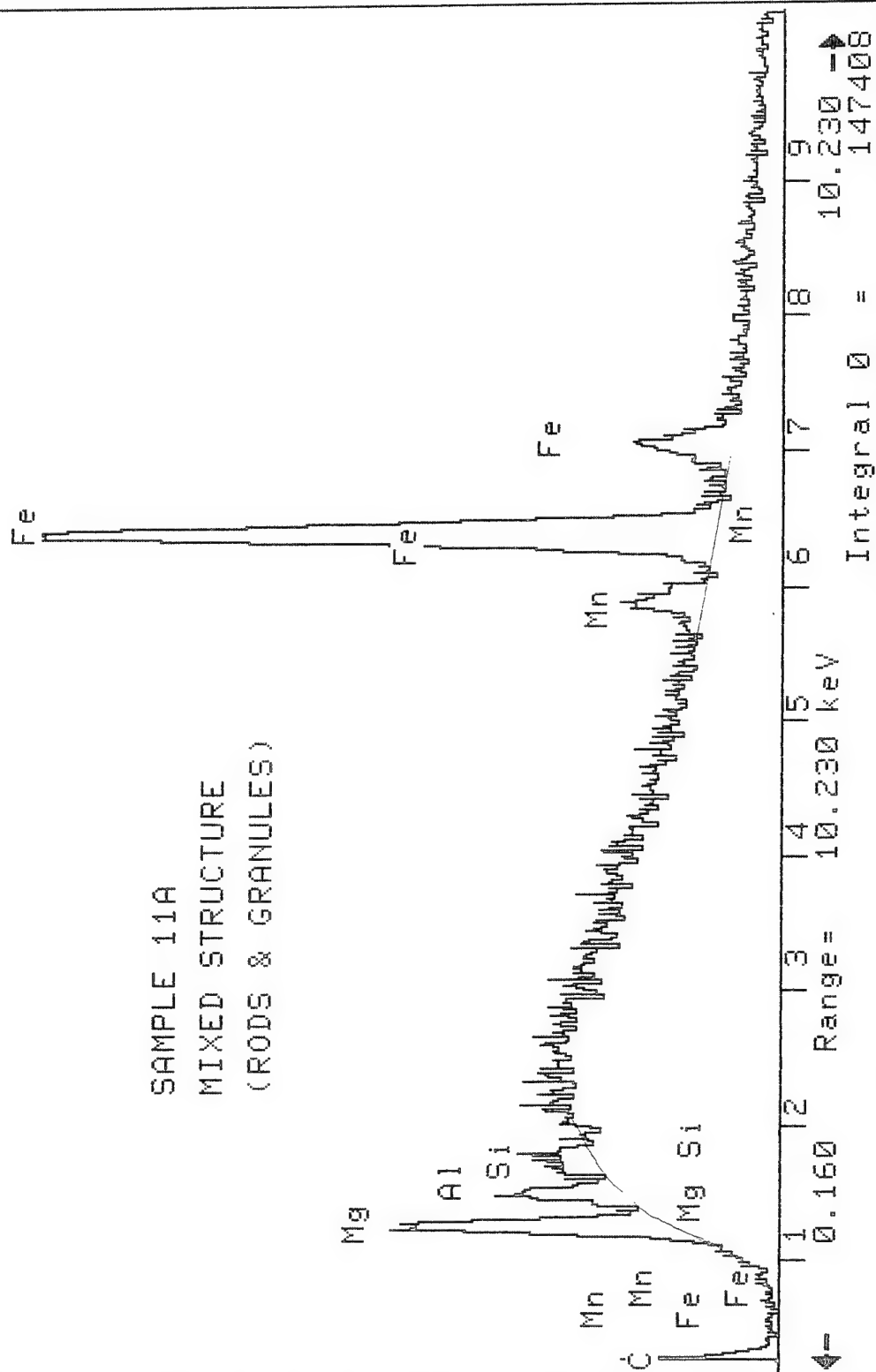
SAMPLE 11A - MIXED STRUCTURE (RODS & GRANULES.)



14-Oct-1992 13:50:14

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 92 secs

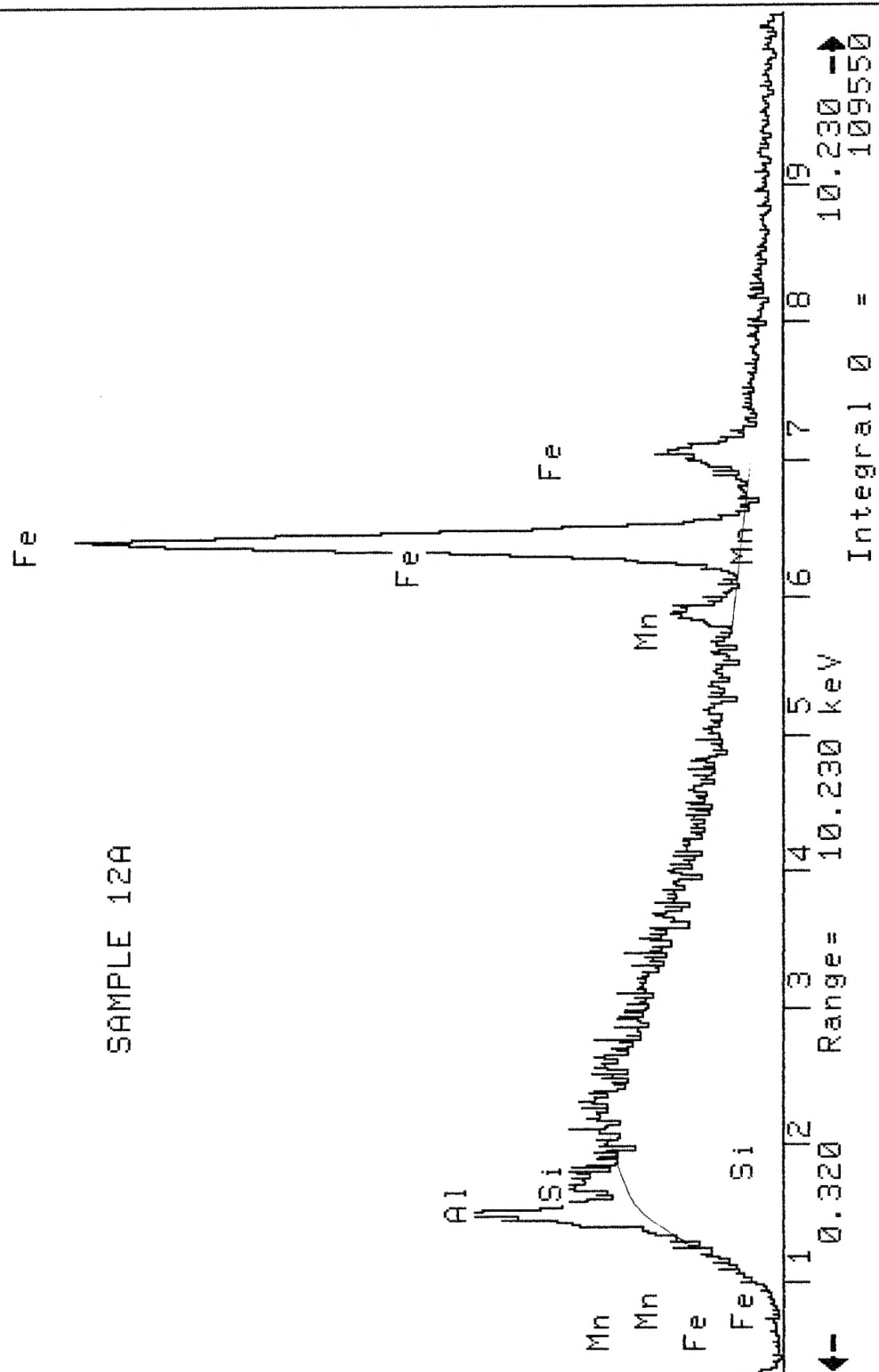
SAMPLE 11A  
MIXED STRUCTURE  
(RODS & GRANULES)



13-Oct-1992 13:59:04

Vert= 1000 counts Disp= 1 Preset= Off Elapsed= 51 secs

SAMPLE 12A









## TECHNOLOGY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security programs, specializing in advanced military space systems. The Corporation's Technology Operations supports the effective and timely development and operation of national security systems through scientific research and the application of advanced technology. Vital to the success of the Corporation is the technical staff's wide-ranging expertise and its ability to stay abreast of new technological developments and program support issues associated with rapidly evolving space systems. Contributing capabilities are provided by these individual Technology Centers:

**Electronics Technology Center:** Microelectronics, VLSI reliability, failure analysis, solid-state device physics, compound semiconductors, radiation effects, infrared and CCD detector devices, Micro-Electro-Mechanical Systems (MEMS), and data storage and display technologies; lasers and electro-optics, solid state laser design, micro-optics, optical communications, and fiber optic sensors; atomic frequency standards, applied laser spectroscopy, laser chemistry, atmospheric propagation and beam control, LIDAR/LADAR remote sensing; solar cell and array testing and evaluation, battery electrochemistry, battery testing and evaluation.

**Mechanics and Materials Technology Center:** Evaluation and characterization of new materials: metals, alloys, ceramics, polymers and composites; development and analysis of advanced materials processing and deposition techniques; nondestructive evaluation, component failure analysis and reliability; fracture mechanics and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures; launch vehicle fluid mechanics, heat transfer and flight dynamics; aerothermodynamics; chemical and electric propulsion; environmental chemistry; combustion processes; spacecraft structural mechanics, space environment effects on materials, hardening and vulnerability assessment; contamination, thermal and structural control; lubrication and surface phenomena; microengineering technology and microinstrument development.

**Space and Environment Technology Center:** Magnetospheric, auroral and cosmic ray physics, wave-particle interactions, magnetospheric plasma waves; atmospheric and ionospheric physics, density and composition of the upper atmosphere, remote sensing using atmospheric radiation; solar physics, infrared astronomy, infrared signature analysis; effects of solar activity, magnetic storms and nuclear explosions on the earth's atmosphere, ionosphere and magnetosphere; effects of electromagnetic and particulate radiations on space systems; space instrumentation; propellant chemistry, chemical dynamics, environmental chemistry, trace detection; atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiative signatures of missile plumes, and sensor out-of-field-of-view rejection.